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RI/FS PLAN
Project No. 60776

Volume 3 of 3
Quality Assurance Project Plan
Appendices G, H, I, J, K & L

Prepared for:

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PRP Group
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April 1990

APPENDIX G

WARZYN FIELD EQUIPMENT PROCEDURES

APPENDIX G-1

FIELD MEASUREMENTS OF pH

pH

Scope and Application: This method is applicable to surface water, wastewater and groundwater.

Method: Potentiometric

Reference: EPA 1983, p. 150.1

Sensitivity: 0.01 pH unit

Optimum Range: pH 1.00 to 12.00

Sample Handling: Determine on-site, if possible

Reagents and Apparatus:

1. pH meter
2. Combination pH electrode.
3. Magnetic stirrer and stir bars
4. Beakers or plastic cups.
5. pH buffer solutions, pH 4.00, 7.00, and 10.00. (Certified buffer solutions)
6. Deionized water in squirt bottle.

Calibration:

1. Place combination electrode in pH 7.00 buffer solution.
2. After allowing several minutes for meter to stabilize, turn calibration dial until reading of 7.00 is obtained.
3. Rinse electrode with deionized water and place in pH 4.00 or pH 10.00 buffer solution. Use pH 7.00 and 4.00 for samples with pH <8, and buffers 7.00 and 10.00 for samples with pH >8.
4. Wait several minutes and then turn slope adjustment dial until reading of 4.00 or 10.00 is obtained.
5. Rinse electrode with deionized water and place in pH 7.00 buffer. If meter reading is not 7.00, follow Steps 2-5 again.

Procedure:

1. Calibrate meter using calibration procedure.
2. Pour the sample into clean beaker or plastic cup.

3. Place stir bar in beaker and put on magnetic stirrer (low speed) for lab measurement of pH. Swirl cup gently for field measurement of pH.
4. Check temperature of sample. It should be $\pm 2^{\circ}\text{C}$ of the buffer solutions.
5. Rinse electrode with deionized water.
6. Immerse electrode in sample. The white KCl junction on side or bottom of electrode must be fully immersed in solution. Allow sufficient time for reading to stabilize. Record pH. Rinse electrode with deionized water.
7. Recheck calibration with pH 7.00 buffer solution after every 20 samples and at the end of the analytical run.

Quality Control:

1. Duplicate 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate is still required. Duplicates should be ± 0.2 pH units. Average the results.
2. All glassware is to be soap and water washed, tap rinsed and deionized water rinsed prior to analyses.

Notes:

1. The pH test is temperature dependent. Therefore, temperatures of buffers and samples should be within 2°C of each other. For refrigerated or cool samples, use refrigerated buffers to calibrate meter.
2. Interferences in pH measurements occur with presence of weak organic and inorganic salts, and oil and grease. If oil and grease are visible, note on data sheet. Clean electrode with soap and water, followed by 10% HCl and deionized water. Then recalibrate meter before analysis of next sample.
3. Electrode should be stored in pH 4.00 buffer.
4. Before leaving laboratory for field work:
 - a. Check batteries.
 - b. Do quick calibration at pH 7.00 and 4.00 to check electrode response and batteries.
 - c. Obtain fresh pH buffer solutions.

5. Following field measurements:

- a. Report any problems with meter or electrode.
- b. Clean meter and meter case.
- c. Make sure electrode is stored in pH 4.00 buffer.

Approved 7/22/86

Michael J. Linsens

Michael J. Linsens
Laboratory Manager

[ALM-1-26]

BECKMAN

Φ^{TM} 10 pH Meter

Φ^{TM} 11 pH Meter

Φ^{TM} 12 pH/ISE Meter

HSSE.

WARRANTY

Your Φ^M (407M, 10, 11, or 12 pH Meter) is warranted to be free of manufacturing defects for one (1) year from the date of purchase. This does not include any defects that are the result of misuse or accident of the instrument. Beckman Instruments, Inc., will, at Beckman's option, repair or replace your instrument with a comparable unit. This is a limited warranty. You may have additional rights under your state laws. Batteries are not included in this warranty.

WARNING: This equipment generates, uses, and can radiate radio frequency energy and may cause interference to radio communications. Improper installation or modification of the equipment may increase interference. It has been tested and found to comply with the limits for Class A computing device products, which are designed to provide reasonable protection against such interference when operated in a controlled environment. Operation of this equipment in a residential area may cause interference, in which case the user at his own expense will be required to take whatever measures may be required to correct the interference.

Beckman Instruments 015-24800-8

BECKMAN

Φ^M 10 pH Meter

Φ^M 11 pH Meter

Φ^M 12 pH/ISE Meter

pH MEASUREMENT			
(Two-standard method; Condensed Instructions)			
I. SETUP			
1 Prepare buffers (eg., pH 4 and 7).	2 Prepare sample.	3 Prepare deionized or distilled water for electrode rinse.	
4 Connect electrodes to instrument.	(1) Omit reference if combination electrode is used. (2) ATC optional.	5 Turn on and clear instrument.	6 Display will read:
II. STANDARDIZE			
1 Rinse electrode(s). Blot excess.	2 Immerse electrode(s) in STD 1. Stir briefly.	3 Press pH , then STD 1 .	4 After STD stops flashing, display will read pH of STD 1.
5 Rinse electrode(s). Blot excess.	6 Immerse electrode(s) in STD 2. Stir briefly.	7 Press STD 2 .	8 After STD stops flashing, display will read pH of STD 2.
III. MEASURE pH			
1 Rinse electrode(s). Blot excess.	2 Immerse electrode(s) in sample. Stir briefly.	3 Press pH .	4 After STD stops flashing, display will read pH of sample.

FOR MORE DETAILED INSTRUCTIONS ON pH MEASUREMENT, SEE NEXT PAGE.

MEASURING mV AND RELATIVE mV (Φ11, Φ12)

MEASURING CONCENTRATION (Φ12)

INSTRUMENT FUNCTIONS AND FEATURES

ELECTRODES, BUFFERS, AND ACCESSORIES

BATTERY REPLACEMENT, SERVICE
AND TROUBLESHOOTING

SPECIFICATIONS

pH MEASUREMENT: DETAILED INSTRUCTIONS

METHODS: The pH 10, 11, and 12 can measure pH from 0 to 15.98. They will perform one- or two-point standardization automatically, using any buffer listed below, at any temperature between -5°C and 100°C.

STANDARD pH BUFFERS RECOGNIZED BY THE pH 10, 11, AND 12:
1.68, 4.00, 7.00, 10.01, 12.45.

TWO-POINT STANDARDIZATION METHOD:

Two-point standardization, the preferred and more accurate method of pH measurement, should be used when pH accuracy of beyond ± 0.1 pH is required. Use buffers as close to the sample pH as possible; one above, and one below. (For example, if sample pH is about 8.5, use 7.00 and 10.01 pH buffers).

ONE-POINT STANDARDIZATION METHOD:

One-point standardization, a somewhat faster procedure, is recommended only if (a), accuracy of ± 0.1 pH unit is acceptable, and (b), sample pH is within 1.5 pH of that of the buffer used for standardization.

pH MEASUREMENT PROCEDURE:

1. Connect electrode(s) to appropriate input(s):
 - a. If a combination electrode is used, connect it to the input marked "pH".
 - b. If an electrode pair is used, connect the indicating electrode to the input marked "pH" and the reference electrode to the input marked "REF".
 - c. For better accuracy, or when measuring and/or standardizing at a temperature of other than 25°C, connect a Beckman 558115 Automatic Temperature Compensator probe to input marked "ATC".
2. Press **[ON]** to turn on instrument, then press **[C]** to clear. Display will show [Clr, AUTO].
3. Rinse electrode(s) (and ATC if used) with deionized water. Blot excess.
4. Immerse electrode(s) (and ATC if used) in first standard. Stir briefly with electrodes to remove bubbles from electrode surfaces. Press **[M]**. Displayed pH value will have a resolution of 0.01. If 0.1 resolution is desired, press **[1]**.
5. Press **[H]**. When [CD] stops flashing, display will show [pH value locked, CD, > st, > st].
6. Rinse electrode(s) (and ATC probe if used) with deionized water. Blot excess. Proceed to appropriate step, according to desired type of standardization:
 - a. If ONE-POINT standardization is to be used, instrument is ready for sample measurement; proceed to Step 9.
 - b. If TWO-POINT standardization is desired, proceed to Step 7.
7. Immerse electrode(s) (and ATC if used) in second standard. Stir briefly with electrodes to remove bubbles from electrode surfaces. Press **[H]**. When [CD] stops flashing, display will show [pH value locked, CD, > st, > st].
8. Rinse electrode(s), (and ATC probe if used) with deionized water. Blot excess.
9. Immerse electrode(s) (and ATC if used) in sample. Stir briefly with electrodes. Press **[M]**. When [CD] stops flashing, display will show [pH value locked, CD]. Measurement is now complete. Repeat Steps 8 and 9, above, for additional samples.
10. If continuous pH monitoring is desired, press **[ON]** to turn off Auto Read function.

MEASURING mV AND RELATIVE mV (Φ11, Φ12)

MEASURING CONCENTRATION (Φ12)

INSTRUMENT FUNCTIONS AND FEATURES

ELECTRODES, BUFFERS, AND ACCESSORIES

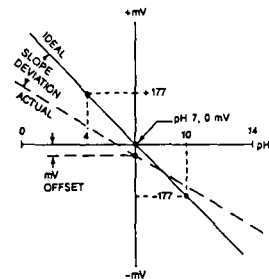
BATTERY REPLACEMENT, SERVICE AND TROUBLESHOOTING

SPECIFICATIONS

pH MEASUREMENT: PRINCIPLES AND THEORY

The pH 10/11/12 pH Meter is essentially a high-impedance voltmeter with a microcomputer that translates voltage and temperature data into pH units. At 25°C, the ideal pH electrode system develops -59 mV per pH unit increase, with 7.00 pH = 0 mV.

Standardization allows the meter to compensate for non-ideal electrode characteristics. One-point standardization compensates for millivolt offset; two-point standardization compensates for both millivolt offset and slope deviation. See diagram below.



Ideal and Actual Electrode Response Compared

The pH 10, 11, and 12 may be standardized with any of five standard pH buffers: 1.68, 4.00, 7.00, 10.01, and 12.45. Standardization may be accomplished with any two buffers, used in any order (and at any temperature, if ATC is used). When [ST] is pressed, the instrument automatically recognizes the buffer. The relationship between pH and electrode voltage changes with temperature. For precise pH measurements or temperatures not close to 25°C, a Beckman 596115 ATC (Automatic Temperature Compensator) probe should be used. With this probe, the instrument automatically compensates for the temperature characteristics of the buffer, permitting a sample to be measured at any temperature, even if different from the buffer temperatures. With ATC, the instrument measures and displays temperatures from -5°C to 100°C.

If an ATC probe is not used, the instrument defaults and displays 25°C.

The pH calculation is based on the Nernst equation:

$$E = E_0 - \frac{2.3 RT}{nF} \log a_i$$

E is the total potential, in millivolts, developed between the sensing and reference electrodes; E_0 varies with the choice of electrodes, temperature, and pressure; $2.3RT/nF$ is the Nernst factor (R and F are constants, n is the charge on the ion, including sign, T is the temperature in degrees Kelvin), and a_i is the activity of the ion to which the electrode is responding.

For further information on principles and theory of pH measurement, refer to The Beckman Handbook of Applied Electrochemistry (Beckman Bulletin 7739).

MEASURING mV AND RELATIVE mV (ϕ11, ϕ12)

MEASURING CONCENTRATION (ϕ12)

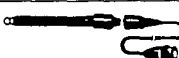
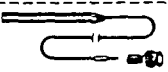
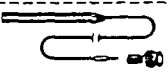
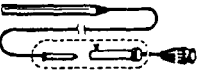
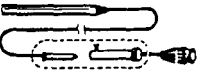


INSTRUMENT FUNCTIONS AND FEATURES


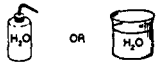


ELECTRODES, BUFFERS, AND ACCESSORIES

BATTERY REPLACEMENT, SERVICE
AND TROUBLESHOOTING

SPECIFICATIONS

FOR ACCURATE mV MEASUREMENTS WITH THE $\Phi 11$ OR $\Phi 12$, THE FOLLOWING ITEMS ARE RECOMMENDED:

1.		pH indicating electrode, Futura II. Futura II cable with BNC connector. NOTE: If combination pH electrode is used, omit separate reference electrode (item 2, below).
OR		
1a.		Metallic electrode with 2 mm pin connector + pin-to-BNC adaptor.
OR		
1b.		Ion-Selective electrode with BNC connector (or U.S. standard connector + U.S. standard-to-BNC adaptor).
2.		Reference electrode, Futura II. Cable with 2 mm pin connector, Futura II. NOTE: Omit reference electrode if combination pH electrode is used.
3.		Standard solution(s) appropriate to the application.

4.		Clean beaker(s) or equivalent container(s), 100-250 mL, for containing standard solution(s).
5.		Squirt bottle or beaker containing deionized or distilled water for rinsing electrodes.
6.		Clean towels, "Kimwipes™", etc., for blotting electrodes.
7.		The sample to be measured.

For part numbers, see "Electrodes, Buffers, and Accessories."
For mV measurement procedures, see next page.

MEASURING mV AND RELATIVE mV ($\Phi 11$, $\Phi 12$)

MEASURING CONCENTRATION ($\Phi 12$)

INSTRUMENT FUNCTIONS AND FEATURES



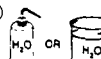



ELECTRODES, BUFFERS, AND ACCESSORIES

BATTERY REPLACEMENT, SERVICE
AND TROUBLESHOOTING





SPECIFICATIONS

mV MEASUREMENT: CONDENSED INSTRUCTIONS



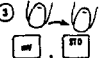





I. SETUP

<p>①</p>  <p>STANDARD</p> <p>Prepare standard solution.</p>	<p>②</p>  <p>SAMPLE</p> <p>Prepare sample.</p>	<p>③</p>  <p>H₂O OR H₂O</p> <p>Prepare deionized or distilled water for electrode rinse.</p>
<p>④</p>  <p>REF pH ATC</p> <p>Connect electrodes to instrument.</p>	<p>⑤</p>  <p>0, C</p> <p>Turn on and clear instrument.</p>	<p>⑥</p>  <p>Display will read:</p> <p>0.00</p>

II. mV MEASUREMENT, ABSOLUTE. FOR RELATIVE mV MEASUREMENT, SEE III, BELOW.

<p>①</p>  <p>H₂O</p> <p>Rinse electrode(s). Blot excess H₂O.</p>	<p>②</p>  <p>STANDARD OR SAMPLE</p> <p>Immerse electrode(s) in standard or sample. Stir briefly.</p>	<p>③</p>  <p>mv</p> <p>Press mv.</p>	<p>④</p>  <p>After mv stops flashing, display will read absolute mV of solution.</p> <p>0.152</p> <p>25.0 °C</p>
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For RELATIVE mV Measurement,
Proceed with following steps:

<h3>III. mV MEASUREMENT, RELATIVE</h3>			
<p>①</p>  <p>H₂O</p> <p>Rinse Electrodes. Blot excess.</p>	<p>②</p>  <p>STANDARD</p> <p>Immerse electrodes in standard solution to be used to establish zero mV point. Stir briefly.</p>	<p>③</p>  <p>mv, STD</p> <p>Press mv then STD.</p>	<p>④</p>  <p>After STD stops flashing, display will read 000.0 mV.</p> <p>REL mV</p> <p>000.0</p> <p>25.0 °C</p>
<p>⑤</p>  <p>H₂O</p> <p>Rinse electrodes. Blot excess.</p>	<p>⑥</p>  <p>SAMPLE</p> <p>Immerse electrodes in sample. Stir briefly.</p>	<p>⑦</p>  <p>mv</p> <p>Press mv.</p>	<p>⑧</p>  <p>After mv stops flashing, display will read mV relative to the standard.</p> <p>REL mV</p> <p>055.4</p> <p>25.0 °C</p>

NOTE
INSTRUMENT WILL REMAIN IN RELATIVE mV MODE UNTIL
EITHER **mv**, **STD**, OR **mv** IS PRESSED.

FOR MORE DETAILED INSTRUCTIONS ON mV MEASUREMENT, PROCEED TO NEXT PAGE.

MEASURING CONCENTRATION (Φ12)

INSTRUMENT FUNCTIONS AND FEATURES

ELECTRODES, BUFFERS, AND ACCESSORIES

BATTERY REPLACEMENT, SERVICE
AND TROUBLESHOOTING

mV MEASUREMENT: DETAILED INSTRUCTIONS

mV MEASUREMENT: TYPICAL USES

Some uses of the mV mode are monitoring chemical reactions, quantifying ions, and determining the oxidizing-reducing potential (ORP) of a given sample. Because such measurements are usually not specific for a particular ion or species, readings must be interpreted carefully to obtain meaningful results. The user should have an understanding of the reaction that is occurring, or is desired, and of any sample components that could potentially interfere. For more detailed information, refer to the Beckman Handbook of Applied Electrochemistry (Beckman Bulletin 7739).

The mV mode may also be used with ion-selective electrodes. The relative mV mode can be used in the standard addition or standard subtraction method of ion analysis.

STANDARD SOLUTION(S)

Make up appropriate standard solution(s) to provide known voltage(s), depending on the reference electrode used and the temperature. For example, common standards used in redox measurements are pH 4 and pH 7 buffers saturated with quinhydrone.

mV MEASUREMENT PROCEDURE

1. Connect electrodes to appropriate inputs:
 - a. Connect indicating electrode to input marked "pH". A Pin-to-BNC Adaptor may be required as most metallic electrodes have a pin connector.
 - b. Connect reference electrode to input marked "REF".
2. Press **[ON]** to turn on instrument, then press **[S]** to clear. Display will show [Cl; ALTO].
3. Rinse electrodes with deionized water. Blot excess.
4. Immerse electrodes in desired solution. Press **[mV]**. Displayed value is absolute mV, as indicated by display of [mV]. When [mV] stops flashing, display will show [mV reading locked, mV].

RELATIVE mV MEASUREMENT PROCEDURE

1. Perform Steps 1 through 3 of mV MEASUREMENT PROCEDURE, above.
2. Immerse electrodes in standard solution to be used to establish the zero mV point. Press **[REL]**, then **[mV]**. When [mV] stops flashing, display will read [000.0 mV]. Note that, in mV mode, pressing **[REL]** causes the instrument to establish the zero mV point at the value of the current reading. If desired, this step may be repeated at any time to re-establish the zero mV point.
3. Rinse electrodes with deionized water. Blot excess.
4. Immerse electrodes in sample. Press **[mV]**. Displayed value is relative mV, as indicated by display of [REL mV]. When [mV] stops flashing, display will show [sample relative mV value locked, mV]. Absolute mV reading of the standard solution is automatically subtracted from the absolute mV reading of the sample, resulting in a relative mV reading for the sample.
5. If continuous readout of relative mV is desired, press **[AUTO]** to turn off Auto Read function.

NOTE

VOLTAGE DIFFERENCE BETWEEN STANDARD SOLUTION AND SAMPLE MUST NOT EXCEED 1000 mV. MAXIMUM DISPLAY RANGE IN mV MODE IS ± 999.9 mV.

NOTE

IN mV MODE, THE 558115 AUTOMATIC TEMPERATURE COMPENSATOR PROBE MAY BE USED FOR TEMPERATURE MEASUREMENT AND DISPLAY, BUT DOES NOT HAVE ANY TEMPERATURE-COMPENSATING EFFECT.

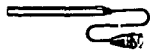
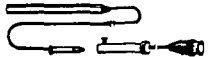


MEASURING CONCENTRATION (§12)


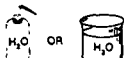


INSTRUMENT FUNCTIONS AND FEATURES

ELECTRODES, BUFFERS, AND ACCESSORIES

BATTERY REPLACEMENT, SERVICE AND TROUBLESHOOTING

FOR ACCURATE CONCENTRATION MEASUREMENTS WITH THE $\Phi 12$, THE FOLLOWING ITEMS ARE RECOMMENDED:

1.  Ion-selective electrode with BNC connector.
OR
 - 1a.  Ion-selective electrode with U.S. standard connector + U.S. standard-to-BNC adaptor.
 2.  Reference electrode, Futura II.
Futura II cable with 2 mm pin connector.
NOTE: Depending on the application, a salt-bridge or double-junction electrode may be required.
 3.  Two standard solutions of appropriate concentration, selected from the following values: 100, 250, 500, 1000, 2500, 5000, 10000, 25000, 50000, and 100000 units. Concentration can be expressed in any desired units such as ppm, mM, mg/L, and $\mu\text{g/L}$.
Make up these solutions per procedure or by diluting a stock solution to suit your requirement.
- NOTE
CONCENTRATION UNITS FOR BOTH STANDARD SOLUTIONS MUST BE THE SAME AS DESIRED FOR SAMPLE READINGS.

4.  Two clean beakers or equivalent containers, approximately 100-250 mL, for containing the two standard solutions.
5.  Squirt bottle or beaker containing deionized or distilled water for rinsing electrodes.
6.  Clean towels, Kimwipes™, etc., for blotting electrodes.
7.  SAMPLE
The sample to be measured.

For part numbers, see "Electrodes, Buffers, and Accessories."
For concentration measurement procedure, see next page.

MEASURING CONCENTRATION ($\Phi 12$)

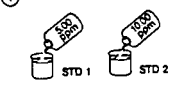

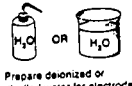

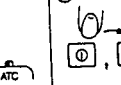
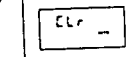
INSTRUMENT FUNCTIONS AND FEATURES

ELECTRODES, BUFFERS, AND ACCESSORIES


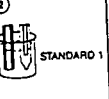

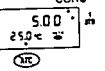



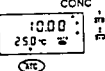
BATTERY REPLACEMENT, SERVICE AND TROUBLESHOOTING

CONCENTRATION MEASUREMENT FOR TWO-STANDARD OPERATION


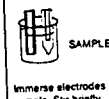

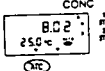
I. SETUP

<p>1</p>  <p>Prepare standard solutions. (e.g., 5.00 and 10.00 units).</p>	<p>2</p>  <p>Prepare sample.</p>	<p>3</p>  <p>Prepare deionized or distilled water for electrode rinse.</p>
<p>4</p>  <p>Connect electrodes to instrument.</p>	<p>5</p>  <p>Turn on and clear instrument.</p>	<p>6</p> <p>Display will read:</p> 

II. STANDARDIZE

<p>1</p>  <p>Rinse electrodes. Blot excess H2O.</p>	<p>2</p>  <p>Immerse electrodes in Standard 1. Stir briefly.</p>	<p>3</p>  <p>Press CONC, then press 1 repeatedly until display shows Standard 1 value, e.g., 5.00.</p>	<p>4</p> <p>When CONC stops flashing, proceed.</p> 
<p>5</p>  <p>Rinse electrodes. Blot excess.</p>	<p>6</p>  <p>Immerse electrodes in Standard 2. Stir briefly.</p>	<p>7</p>  <p>Press 2 repeatedly until display shows Standard 2 Value, e.g., 10.00.</p>	<p>8</p> <p>When CONC stops flashing, proceed.</p> 

III. MEASURE CONCENTRATION

<p>1</p>  <p>Rinse electrodes. Blot excess.</p>	<p>2</p>  <p>Immerse electrodes in sample. Stir briefly.</p>	<p>3</p>  <p>Press CONC.</p>	<p>4</p> <p>After CONC stops flashing, display will read concentration of sample.</p> 
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FOR MORE DETAILED INSTRUCTIONS ON CONCENTRATION MEASUREMENT, PROCEED TO NEXT PAGE.

INSTRUMENT FUNCTIONS AND FEATURES

ELECTRODES, BUFFERS, AND ACCESSORIES

REPLACEMENT SERVICE

CONCENTRATION MEASUREMENT: DETAILED INSTRUCTIONS

The following procedure, utilizing two-point standardization, can be used to measure concentrations of ions in almost any desired units.

STANDARD SOLUTIONS:

Standards can be made from any type of solution, with concentrations selected from the following values: 1.00, 2.50, 5.00, 10.00, 25.0, 50.0, 100.0, 250.0, 500, and 1000 units.

Units of concentration may be any that the user finds convenient. CONCENTRATION UNITS FOR BOTH STANDARD SOLUTIONS MUST BE THE SAME AS DESIRED FOR SAMPLE READINGS.

Some examples of units are: parts per million, percent, moles per liter, parts per billion, milliequivalents per liter, and ounces per gallon.

Select two standard values as close as possible to the anticipated sample value, preferably with one standard value below and one standard value above the sample. For example, if sample solution is about 150 millimoles per liter (mM), make up standards of 100 mM and 250 mM. If sample concentration varies widely, for example, between 10 mM and 75 mM, make up standards of 10 mM and 100 mM.

Standards and samples should be at the same temperature to avoid temperature-dependent variations in readings.

NOTE

Standard and sample solutions may require ionic strength adjustment or interfering ion removal. Consult electrode instructions for details.

CONCENTRATION MEASUREMENT PROCEDURE:

1. Connect electrodes to appropriate inputs:
 - a. Connect ion-selective electrode to input marked "ISE".
 - b. Connect reference electrode to input marked "REF".

NOTE

If, in addition to the ion-selective electrode, a combination pH electrode is connected to the instrument AND is to be immersed in the same solution, DO NOT use a separate reference electrode.

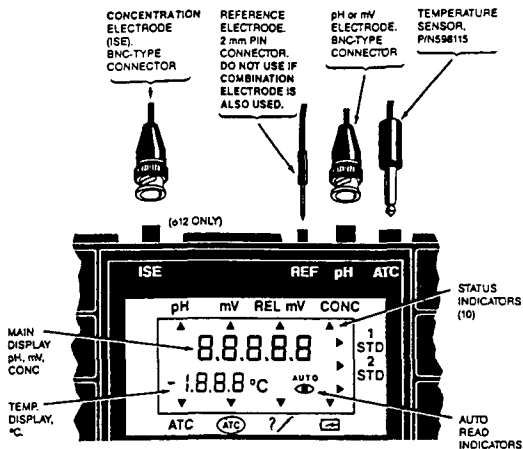
2. Press **[ON]** to turn on instrument, then press **[C]** to clear. Display will show [Ck AUTO].
3. Rinse electrodes with deionized water. Blot excess.
4. Immerse electrodes in first standard solution. Press **[MODE]**, then press **[STANDARD]** as many times as needed for the concentration value of the first standard to show on the display. When **[STANDARD]** is pressed repeatedly, the display steps through the following values: 1.00, 2.50, 5.00, 10, 25, 50, 100, 250, 500, and 1000 concentration units. For example, if the concentration of the standard is 100 units, press **[STANDARD]** seven times and the display will show [100]. When [**CD**] stops flashing, display will show [100 locked, **CD**].
5. Rinse electrodes with deionized water. Blot excess.
6. Immerse electrodes in second standard solution. The first and second standards must be different, but can be measured in any order. Press **[STANDARD]** as many times as needed for the display to show the concentration value of the second standard, e.g., 250. When [**CD**] stops flashing, display will show [250 locked, **CD**].
7. Rinse electrodes with deionized water. Blot excess.
8. Immerse electrodes in sample. Press **[MODE]**. When [**CD**] stops flashing, display will show [sample value locked, **CD**]. Measurement is now complete. Repeat Steps 7 and 8, above, for additional samples.
9. If continuous concentration readout is desired, press **[HOLD]** to turn off Auto Read function.

INSTRUMENT FUNCTIONS AND FEATURES

ELECTRODES, BUFFERS, AND ACCESSORIES

BATTERY REPLACEMENT, SERVICE

ELECTRODE CONNECTIONS



DISPLAY FEATURES AND STATUS INDICATORS

DISPLAY

The large digits show the following:

1. Readout of the measured variable: pH, mV, or concentration.
2. [Clr] is displayed, indicating that instrument is cleared, when [E] is pressed.
3. Error message:

[Er] indicates an excessive, potentially damaging, input voltage, typically caused by static electricity when the electrode pair is not in solution. In this case, immerse electrodes in solution, press [E], and proceed with measurement. If [Er] again appears, check connections and electrodes for possible open circuit.

Temperature Display The small digits display temperature in °C. Will read 25°C if ATC not plugged in.

AUTO

AUTO READ ON/OFF Indicator for AUTO READ ON/OFF key, described subsequently.

AUTO READ Status Indicator (eye symbol). Functions during standardization and when instrument is in AUTO mode. During standardization, the eye symbol starts flashing when [STD] is pressed, and locks on when the reading has stabilized. During sample measurement in AUTO mode, the eye symbol starts flashing when a mode key is pressed, and locks on when the reading has stabilized. The reading remains locked until a mode key is pressed. If an interval of approximately 30 minutes elapses without a key being pressed, the instrument turns off automatically to conserve the batteries, but retains all standardization data in memory.

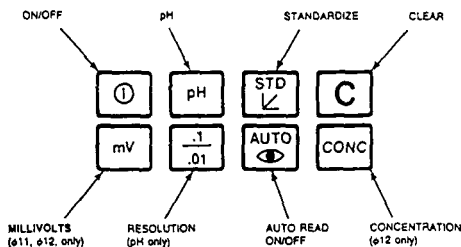
STATUS INDICATORS

- ▼ ATC Indicates that ATC is plugged in. The instrument measures and displays temperature within the range of -5°C and 100°C. Display of [Er] indicates that the temperature sensed is outside the measurement range, or the ATC is nonfunctional.
- ▼ ATC Indicates that ATC is not plugged in. The temperature reading defaults to 25°C.
- ▲ pH Indicates that instrument is in pH mode.
- ▲ mV Indicates that instrument is in mV mode.
- ▲ REL mV Indicates that instrument is in relative mV mode.
- ▲ CONC Indicates that instrument is in concentration mode, ø12 only.
- ▲ 1 STD Indicates that one standard has been used to standardize for the selected measurement mode (pH or CONC).
- ▲ 2 STD Indicates that two standards have been used to standardize for the selected measurement mode (pH or CONC).
- ▼ 7 Indicates a questionable electrode and/or standardization.
- ▼ 7 Indicates that batteries should be replaced.

INSTRUMENT FUNCTIONS AND FEATURES

ELECTRODES, BUFFERS, AND ACCESSORIES

KEYPAD



KEYPAD FUNCTIONS

KEY

- ① **Instrument ON/OFF Key.** When OFF, the instrument retains the standardization data in memory. Instrument shuts off automatically after 30 minutes of inactivity if AUTO READ is ON. (See below.)
- C **Clear Key.** Clears instrument, resetting all standardization data to default values, and returning instrument to AUTO Mode.
- AUTO
👁️ **Auto Read Key.** Turns Auto Read function ON and OFF.
 1. When Auto Read is ON:
 - a. The word [AUTO] appears on the display.
 - b. The instrument tests the electrode signal for stability. During this test, [👁️] flashes ON and OFF. When the signal has met the stability requirement (see SPECIFICATIONS), [👁️] remains on continuously, and the digital display locks onto the reading. No further measurements are made until a key is pressed.
 - c. After 30 minutes without keypad input, the instrument turns off automatically but retains all standardization data.
 2. When Auto Read is OFF:
 - a. [AUTO] disappears from display.
 - b. The instrument continuously measures and displays in the selected mode: pH, mV, or CONC.
 - c. After 1 to 2 hours without keypad input, the instrument turns off automatically but retains all standardization data.
- .1
—
.01 **pH Resolution Selection Key.** Changes resolution of the displayed pH reading from 0.01 to 0.1 pH unit, or vice versa. At the lower resolution (0.1), time required for the Auto Read to lock is shorter. (See SPECIFICATIONS).
- pH **Selects the pH measurement mode.**
- mV **Selects the mV mode (pH1, pH2), for measurement of either absolute or relative millivolts. See MEASURING mV AND RELATIVE mV.**
- CONC **Selects the concentration measurement mode (pH2). Used with specific ion electrodes.**
- STD
↙ **Standardize Key.** Standardizes instrument. Depends upon mode.
 - a. pH Mode: [STD] Key causes the instrument to automatically identify the pH value of the buffer from any one of the following: 1.68, 4.00, 7.00, 10.01, and 12.45.
 - b. mV Mode (pH1, pH2): [STD] Key causes the instrument to establish the zero-millivolt level at the value of the current reading. Instrument is now in Relative mV mode.
 - c. CONC Mode (pH2): Repeated pressing of [STD] Key causes the instrument to step through the following sequence of values: 1.00, 2.50, 5.00, 10, 25, 50, 100, 250, 500, and 1000 concentration units.

1. FUTURA II ELECTRODES

COMBINATION ELECTRODES:

	Standard	Probe	Test-Tube
Glass Body Ag/AgCl, Refillable	39590	8-10" x 3/8"	39522
Glass Body Calomel, Refillable	39527	39521	39522
Epoxy Body Calomel, Refillable	39639	39528	39525, 39526 (7")
Epoxy Body Ag/AgCl, Refillable	39631	39633	39635
Epoxy Body Ag/AgCl, Gel Filled	39636	39632	39634
Epoxy Body, Star Ag/AgCl Refillable	39637		
Glass Body, Star Ag/AgCl Refillable	39524		
Flat Bulb, Epoxy Body	39523		

ELECTRODE PAIRS:

pH INDICATING ELECTRODES:

0-14 pH, Spherical Bulb	39314
0-11 pH, Dome Bulb (Durable)	39316

METALLIC ELECTRODES:

Silver Billet	39261
Platinum Inlay	39273

REFERENCE ELECTRODES:

Calomel Half Cell, Quartz Fiber Junction	39416
Calomel Half Cell, Ceramic Frit Junction	39417
Ag/AgCl Half Cell, Quartz Fiber Junction	39418
Calomel Half Cell, Sleeve Double Junction	39419
Calomel Half Cell, Inverted Sleeve Junction	39420
Ag/AgCl Half Cell, Inverted Sleeve Junction	39421

2. FUTURA II KEEPER CABLES

COMBINATION AND INDICATING ELECTRODE CABLES

1m, BNC Connector	59757B
2m, BNC Connector	59757B
6m, BNC Connector	597580

REFERENCE ELECTRODE KEEPER CABLES

1m, 2mm Pin Connector	598982
2m, 2mm Pin Connector	598983
6m, 2mm Pin Connector	598984

3. SALT BRIDGE: 563853

4. SUBMERSIBLE COMBINATION pH ELECTRODE WITH ATC: 39530

5. AUTOMATIC TEMPERATURE COMPENSATOR, 598115:

Permits temperature measurement and display, and temperature compensation of pH and ion-selective electrodes, within range of -5°C to 100°C. Epoxy body. For use with standard 5-inch (13-cm) electrodes. Includes 39" (1 meter) cable with miniature phone jack.

6. ELECTRODE ADAPTORS:



592362 Standard to
BNC Adaptor



592367 PIN to BNC Adaptor

Adapts Glass Electrode (GE) BNC terminal on
pH Series pH Meters to accommodate
electrodes with U.S. Standard Connectors.

Adapts Glass Electrode (GE) BNC terminal on
pH Series pH Meters to accommodate
electrodes with PIN Connectors.
Typically used to connect metallic electrodes.

7. BUFFERS

	6 Pack of Pints	1 Gallon	5 Gallons	Powder (Colorless)
pH 4 Buffer (red)	582517	566001	582822	3005
pH 7 Buffer (green)	582521	566003	582823	3007
pH 10 Buffer (blue)	582525	566005	582824	3019
pH 12.45				3010

8. FILLING SOLUTIONS

Description	Quantity	Part No.
Combination Electrode Filling Solution or Ag/AgCl Reference Electrode Filling Solution (4M KCl/AgCl saturated; to be used with Ag/AgCl internals)	4-pack of 100 mL bottles	566467
Reference Electrode Filling Solution (saturated KCl to be used with Calomel internals)	4-pack of 100 mL bottles	566468
Electrode Soaking Solution	4-pack of 100 mL bottles	566576
Salt Bridge Solution, Contains Sodium Nitrate and Sodium Acetate	4-pack of 100 mL bottles	566469
Filling Solution, 1M, KCl Saturated with AgCl (Star-Series electrodes only)	4-pack of 100 mL bottles	598943

9. pH START-UP KIT:

39831 Electrode, Cable, Thermocompensator, Sample Buffers, Filling solution	123135
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10. pH STAND LAB ORGANIZER

	123136
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11. pH DELUXE FIELD CASE

	123128
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12. pH SOFT CASE

	123127
--	--------

13. pH MOUNT, WALL/SHELF BRACKET

	599190
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
Your 410, 11, or 12 is powered by two 3.6 volt lithium batteries. Expected battery life is over 1,000 hours of continuous operation. Replacement batteries can be obtained by ordering Part No. 945754 from your local Beckman office. (In U.S. call 1-800-742-2345).

Acceptable replacement batteries are also available on a world-wide basis:

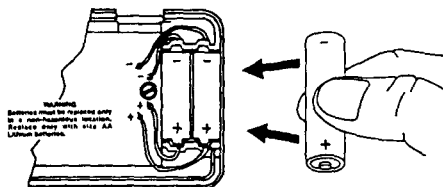
Mfr.	Part No.
Electrochem Industries	3B840-TC
Power Conversion Inc.	106-41
Salt Advanced Battery Div.	LS8
Tadiran	TL-2100 AA/S

Local suppliers may be found in your telephone directory.


Note that these batteries are 3.6 volt lithium cells. Do not attempt to replace them with 1.5 volt alkaline or carbon-zinc cells.

If instrument display indicates low battery voltage  or if display is blank when instrument is turned on, batteries should be replaced:



1. Remove 2 Phillips screws and bottom cover from instrument.
2. Lift out old batteries.
3. Note (+) and (-) markings in battery compartment.
4. Check (+) and (-) markings on batteries and insert as shown:

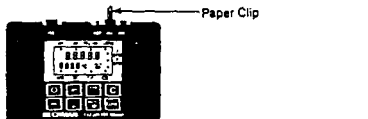




5. Replace back cover and screws.

IMPORTANT: A "BREAK-IN" PERIOD OF UP TO 30 MINUTES IS REQUIRED WHEN SOME NEW LITHIUM BATTERIES ARE FIRST PLACED INTO SERVICE. DURING THIS PERIOD, THE LOW BATTERY SYMBOL AND SOME "GHOSTING" MAY APPEAR ON THE DISPLAY. IF SO, LEAVE INSTRUMENT ON FOR 20-30 MINUTES AND THEN PRESS . THE LOW BATTERY SYMBOL AND "GHOSTING" SHOULD DISAPPEAR.




TROUBLESHOOTING PROCEDURE

1. Disconnect electrode cable(s) from instrument. Press  to turn on instrument, then press  to clear. Display should show [CH AUTO]. If not, replace batteries per BATTERY REPLACEMENT, above. If instrument is still inoperative, call Service Hot Line: 1-800-662-6217.
2. Insert one end of a paper clip into the small hole in the center of the "pH" input connector. Hold the other end of the clip to the inside barrel of the same connector as shown.



3. Press  then . The display should lock at pH 7.00, indicating a one-point standardization. Reading should drift.
4. Reconnect pH electrode cable to "pH" input connector. Short the input connector of the cable.



Press  and then . Display should lock at pH 7.00. Press  then remove paper clip. Reading should drift.

- a. If instrument passes test, go to Step 6.
 - b. If instrument fails test, call Beckman Electrochemistry Applications: 1-800-854-8067 Outside California 714-871-4848 Within California
5. Reconnect pH electrode(s). Immerse electrode(s) in pH 4 buffer and perform one-point standardization. Then immerse electrode(s) in pH 10 buffer and take pH reading. At 25°C, the reading should be between 9.7 and 10.1 pH.
 - a. If the test is passed, the pH meter, cable, and electrode(s) are functioning properly.
 - b. If test is failed, the pH electrode(s) must be rejuvenated or replaced. The electrode rejuvenation

INSTRUMENT BECKMAN PART NO.	pH 10 123132	pH 11 123133	pH 12 123134
pH MEASUREMENT			
Range	0 to 15.99 pH	0 to 15.99 pH	0 to 15.99 pH
Resolution (Selectable)	0.01, 0.1 pH unit	0.01, 0.1 pH unit	0.01, 0.1 pH unit
Relative Accuracy	± 0.01 pH	± 0.01 pH	± 0.01 pH
Auto Read Mode	0.1 pH Resolution: Display locks after reading is stable within 1.0 mV for 4 seconds. 0.01 pH Resolution: Display locks after reading is stable within 0.5 mV for 8 seconds.		
Buffers Recognized by Instrument: 1.68, 4.00, 7.00, 10.01, 12.45 pH.			
MILLIVOLT MEASUREMENT			
Range	—	-999.9 to +999.9 mV	-999.9 to +999.9 mV
Resolution	—	0.1 mV	0.1 mV
Accuracy	—	± 0.2 mV ± 0.02% of reading, relative mV	± 0.2 mV ± 0.02% of reading, relative mV
Auto Read Mode	—	Display locks after reading is stable within 0.5 mV for 8 seconds.	
CONCENTRATION MEASUREMENT			
mV Accuracy	—	—	± 0.1 mV
Auto Read Mode	—	—	Display locks after reading is stable within 0.25 mV for 8 seconds.
Useable Standard Values	—	—	Two values, in any desired units, selected from the following: 1.0, 2.5, 5.0, 10, 25, 50, 100, 250, 500, and 1000.

TEMPERATURE MEASUREMENT (ALL MODELS)	
Range: -5°C to 100°C	
Resolution: 0.1°C	
Accuracy (with Beckman 596115 Probe): ± 0.5°C	
MISCELLANEOUS (ALL MODELS)	
Input Connections:	
1. BNC input for pH, mV, and concentration.	
2. 2-mm pin connector for reference electrode.	
3. Miniature phone jack for Beckman 596115 Automatic Temperature Compensator.	
Operating Temperature: 15°C to 40°C, ambient, non-condensing.	
Power Source: Two lithium cells, 3.6 volts each, AA Size.	
Error Indications:	
1. Input overvoltage (all modes)	
2. Temperature compensation non-functional	
3. Low batteries	
4. Questionable electrode/standardization.	
Size: 5.2 inches x 3.8 inches x 1.3 inches.	

Beckman Co
National
7 West Poplar Ave
-ile, CT 93257

APPENDIX G-2

FIELD MEASUREMENT OF SPECIFIC CONDUCTANCE AND TEMPERATURE

FIELD MEASUREMENT OF SPECIFIC CONDUCTANCE
AND TEMPERATURE

Method: Specific Conductance, umhos @ 25°C

Reference: EPA 1979, Page 120.1, Standard Methods, 15th edition, pp 70-73

Detection Limit: 1 umho/cm @ 25°C

Optimum Range: 0.1 - 100,000 umhos/cm

Sample Handling: Determine on-site or within 24 hours

Reagents and Apparatus:

1. Conductivity meter (YSI) and electrodes.
2. Deionized water in squirt bottle.
3. Standard potassium chloride solution, 0.0100 N.

Procedure:

YSI Conductivity Meter

1. With mode switch at off position, check meter zero. If not zeroed, use meter screw and adjust to zero.
2. Plug probe into jack on side of meter.
3. Turn mode switch to red line, and turn red line knob until needle aligns with red line on dial. Change batteries if cannot be aligned.
4. Totally immerse probe in sample. Do not allow the probe to touch the sample container.
5. Turn mode switch to appropriate conductivity scale, X100, X10, or X1. Use a scale that will give a mid-range output on the meter.
6. Wait for needle to stabilize (about 15 sec.) and record conductivity multiplying by scale setting.
7. While gently agitating the probe, take sample temperature (°C) and record.
8. Rinse probe with deionized water.
9. Record specific conductivity (1st column) and temperature on F.O.S. sheet.

Notes:

1. Calculate conductivity using following formula:

$$G_{25} = \frac{G_T}{[1 + 0.02 (T-25)]}$$

G_{25} = Conductivity at 25°C, umhos/cm

T = Temperature of sample, °C

G_T = Conductivity of sample at temperature T , umhos/cm

2. Report results for the standard solution with each data set.
3. Record on field sheet which meter and probe were used. Meter should be wiped clean as necessary.
4. After returning to lab, compare results with previous data. Report problems to lab personnel.

Reagent Preparation:

1. Stock Potassium Chloride Solution, 1.00 N: Dissolve 74.555 g. KCl in Milli-Q water and dilute to 1,000 ml. in a volumetric flask.
2. Standard Potassium Chloride Solution, 0.0100N: Dilute to 10.0 mls. of stock solution to 1,000 mls. with Milli-Q water using a volumetric pipet and flask.

YSI MODELS 33 AND 33M S-C-T METERS INSTRUCTIONS



GENERAL DESCRIPTION

The YSI Model 33 and 33M S-C-T Meters are portable, battery powered, transistorized instruments designed to accurately measure salinity, conductivity and temperature. They use a probe consisting of a rugged, plastic conductivity cell and a precision YSI thermistor temperature sensor combined in a single unit.

Conductivity with the Model 33 is expressed as microhmhos/centimeter; with the 33M, it is millisiemens/meter (mS/m). These are measurements of the electrical conductance the sample would show if measured between opposite faces of a 1 cm cube. (Conversion information: 1 microhmho/cm = 0.1 mS/m.) Salinity is the number of grams of salt/kilogram of sample (ppt = parts per thousand). This measurement assumes the sample contains a "standard" sea water salt mixture. The sample temperature is measured in degrees Celsius.

Salinity measurements are manually temperature compensated by direct dial. Conductivity measurements are not temperature compensated; however, a temperature function is provided on the instrument to aid with calculation of corrections. When temperature and conductivity are known, it is possible to calculate salinity; and when only temperature and salinity are known, it is possible to calculate conductivity. This is discussed in the section on Recalibration.

SPECIFICATIONS

Model 33 Conductivity

Ranges: 0 to 500 ($\times 1$), 0 to 5,000 ($\times 10$), and 0 to 50,000 microhmhos/cm ($\times 100$) with YSI 3300 Series Probes. (Note: The "microhmho" designations on the meter are a shorthand form for "microhmho/cm".)

Accuracy: (See Error Section)
 $\pm 2.5\%$ max. error at 500, 5,000 and 50,000 plus probe.
 $\pm 3.0\%$ max. error at 250, 2,500 and 25,000 plus probe.

Readability:

2.5 microhmhos/cm on 500 microhmho/cm range.
25 microhmhos/cm on 5,000 microhmho/cm range.
250 microhmhos/cm on 50,000 microhmho/cm range.

Temperature Compensation: None.

Model 33M Conductivity

Ranges:

0 to 50 ($\times 1$), 0 to 500 ($\times 10$), and 0 to 5,000 ($\times 100$) mS/m with YSI 3300 Series Probes.

Accuracy: (See Error Section)

$\pm 2.5\%$ max. error at 50, 500, and 5,000 plus probe.
 $\pm 3.0\%$ max. error at 25, 250, and 2,500 plus probe.

Readability:

0.25 mS/m on 50 mS/m range.
2.5 mS/m on 500 mS/m range.
25.0 mS/m on 5,000 mS/m range.

Temperature Compensation: None.

Salinity

Range: 0-40 ppt in temperature range of -2 to $+45^{\circ}\text{C}$, within specified conductivity range of 0 to 50,000 microhmho/cm (0 to 5,000 mS/m). See chart in section on Recalibration.

Accuracy (See Error Section)

Above 4°C : ± 0.9 ppt at 40 ppt and ± 0.7 ppt at 20 ppt plus conductivity probe.

Below 4°C : ± 1.1 ppt at 40 ppt and ± 0.9 ppt at 20 ppt plus conductivity probe.

Readability: 0.2 ppt on 0-40 ppt range.

Temperature Compensation: Manual by direct dial from -2 to $+45^{\circ}\text{C}$.

Temperature Range: -2 to $+50^{\circ}\text{C}$.

Accuracy: $\pm 0.1^{\circ}\text{C}$ at -2°C , $\pm 0.6^{\circ}\text{C}$ at 45°C plus probe (See Error Section)

Readability: $\pm 0.15^{\circ}\text{C}$ at -2°C to $\pm 0.37^{\circ}\text{C}$ at 45°C .

Power Supply: Two D-size alkaline batteries, Eveready E95 or equivalent, provide approximately 200 hours of operation.

Instrument Ambient Range: -5 to $+45^{\circ}\text{C}$. A maximum error of $\pm 0.1\%$ of the reading per $^{\circ}\text{C}$ change in instrument temperature can occur. This error is negligible if the instrument is readjusted to redline for each reading.



YSI Scientific

Yellow Springs, Ohio 45387 USA • Phone 513 767-7241 • 800 343-HELP

Salinity Error

The salinity readings are a function of temperature and conductivity, therefore the accuracy is a function of both.

The temperature scale and temperature control have been designed to minimize the temperature error contribution to the salinity error. The error shown in Figure 3 is the total of the temperature and conductivity probe, the temperature scale and the salinity scale error.

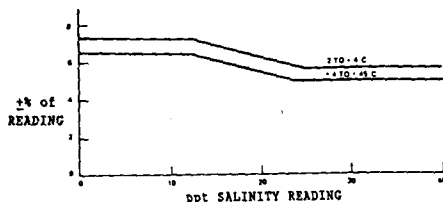


FIGURE 3

Example
Meter Reading: 10 ppt. @ 10°C
% of Reading Error: 6.5%
Accuracy: 10 ppt ± 0.65 ppt for all errors,
combined worst case.

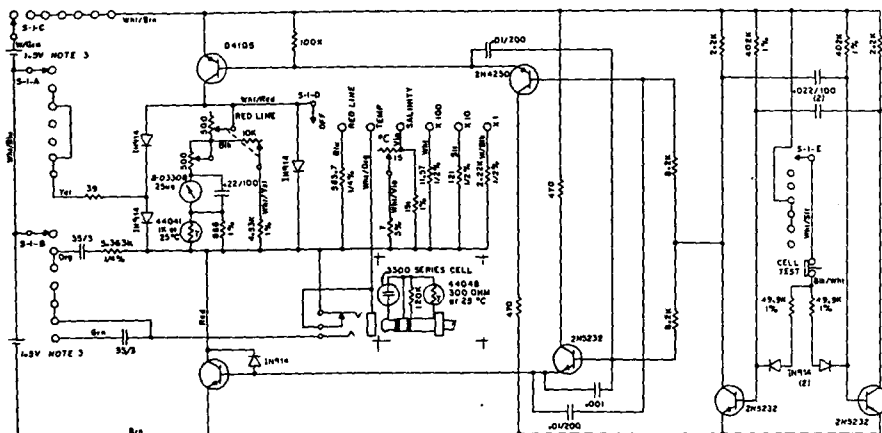
CIRCUIT DESCRIPTION

The circuit is composed of two parts; a multivibrator and switching transistors. The multivibrator produces a square waveform voltage. The square wave is applied to two switching transistors. They alternately apply two batteries of opposite polarity to the probe thus providing AC power which minimizes polarization effects. The meter is in series with one battery and measures the current from it. The current from the battery is proportional to the conductance of the cell. Salinity is measured in a special range conductivity circuit which includes a user-adjusted temperature compensator. In the temperature, redline and X1 positions, the multivibrator operates at 100Hz. In the salinity, X100 and X10 positions the multivibrator operates at 600Hz; in these ranges, pushing the CELL TEST button drops the frequency to 100Hz, allowing the operator to test for probe polarization.

INSTRUMENT MAINTENANCE

The only maintenance required is battery replacement. Two "D" size alkaline flashlight cells, such as Eveready E95 or equivalent, will provide 200 hrs. of operation. Accuracy will not be maintained if zinc-carbon "D" cells are used. Battery replacement is indicated when the redline adjustment cannot be accomplished.

Replace batteries every six months to reduce the danger of corrosion due to leaky batteries. To replace batteries, remove the screws from the rear cover. The battery holders are color coded. The positive end must go on red.



NOTES:

Resistance values in ohms. K = 1,000. Resistors are 1/2 W, 10%, unless otherwise specified.

Battery is D size, alkaline only. Eveready E-95 or equivalent.

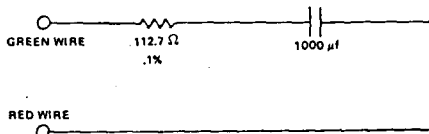
This schematic is representative and may be slightly different from the circuit in your instrument.

2. You may use the resistor and capacitor hookup shown in the sketch to substitute for the probe in the following recalibration procedure.

a. Set the instrument for a salinity measurement as normal.

b. Substitute a 1000 microfarad capacitor and 112.7 ohm 0.1% tolerance resistor for the probe.

Connect the resistor and capacitor between the green wire and red wire on the jack connections inside the instrument.



c. Turn the temperature dial until the meter reads redline.

d. Reinstall the temperature knob with the arrow at 25°C.

This is a temporary calibration only. Return the instrument to the factory for proper recalibration.

YSI 3300 SERIES CONDUCTIVITY/TEMPERATURE PROBES

Description

These probes are designed and constructed for rugged, accurate service in field use. The conductivity cell constant is 5.0/cm (500.0/m) $\pm 2\%$. Each probe contains a precision YSI thermistor temperature sensor of $\pm 0.1^\circ\text{C}$ accuracy at 0°C and $\pm 0.3^\circ\text{C}$ at 40°C . The low capacitance cable assembly terminates in a three terminal 0.25" dia. phone plug.

The 3310 has a 10 foot cable and the 3311 a 50 foot cable. Other lengths are available on special order.

The probe has a rigid P.V.C. body, platinized pure nickel electrodes, and a rugged cable.

Cleaning and Storage

Cleaning

When the cell test indicates low readings the probable cause is dirty electrodes. Hard water deposits, oils and organic matter are the most likely contaminants.

For convenient normal cleaning soak the electrodes for 5 minutes with a locally available bathroom tile cleaning preparation such as Dow Chemical "Bathroom and Chrome Cleaner," Johnson Wax "Envy, Instant Cleaner," or Lysol Brand "Bath, Tub, Tile Cleaner."

For stronger cleaning a 5 minute soak in a solution made of 10 parts distilled water, 10 parts isopropyl alcohol and 1 part HCl can be used.

Always rinse the probe thoroughly in tap water, then in distilled or deionized water after cleaning and before storage.

CAUTION: Do not touch the electrodes inside the probe. Platinum black is soft and can be scraped off.

If cleaning does not restore the probe performance, replatinizing is required.

Storage

It is best to store conductivity cells in deionized water. Cells stored in water require less frequent platinization. Any cell that has been stored dry should be soaked in deionized water for 24 hours before use.

Replatinization

1. Clean the probe.

2. Place the cell in a 50 ml (approximate) jar or beaker and add enough YSI 3140 Platinizing Solution to cover the electrodes. Do not cover the top of the probe.

3. Plug the probe into the Model 33 or 33H, switch to the X100 scale to platinize the electrodes. Move the probe slightly to obtain the highest meter reading and continue platinizing for the approximate time shown below:

Meter Reading microhm/cm	mS/m	Time in minutes
30,000	3,000	5
25,000	2,500	6
20,000	2,000	8
15,000	1,500	11
10,000	1,000	16

4. After the elapsed time, remove the probe and rinse in tap water, then in distilled or deionized water.

5. Return the solution to its container. 2 oz. of solution should be sufficient for 50 treatments.

Probe Use and Precautions

1. Obstructions near the probe can disturb readings. At least two inches of clearance must be allowed from non-metallic underwater objects. Metallic objects such as piers or weights should be kept at least 6 inches from the probe.

2. Weights are attached to the cable of the YSI 3310 and 3311 Probes. The YSI 3327 Weights are supplied in pairs with a total weight of 4 ounces per pair. Should it become necessary to add more weight to overcome water currents, we suggest limiting the total weight to two pounds (8 pairs). For weights in excess of two pounds use an independent suspension cable. In either case, weights must be kept at least 6 inches away from the probe.

3. Gentle agitation by raising and lowering the probe several times during a measurement insures flow of specimen solution through the probe and improves the time response of the temperature sensor.

Conductivity and Salinity Corrections for Long Cables

The additional length of wire in long cables adds capacitance and resistance which will effect readings. The recommended way to correct for these influences is by use of YSI Conductivity Calibrator Solutions (see below), which will permit an estimate of correction factors. If these solutions are not available, the following tables can be used for the correction of errors caused by cable resistance and capacitance on special length versions of the 3310, 3311, S-17933 and S-16120 probes.

APPENDIX G-3

FIELD MEASUREMENT OF DISSOLVED OXYGEN

YSI DISSOLVED OXYGEN METER AND PROBE

Detection Limit: 1.00 mg/L Dissolved Oxygen

Working Concentration Range: 0 - 15 mg/L Dissolved Oxygen

YSI DISSOLVED OXYGEN METER AND PROBE

Scope and Application: The instructions outlined below are to be followed for the daily calibration and routine operation of the YSI Dissolved Oxygen Meter and Probe.

Reference: Instruction Manual YSI Model 54ARC Dissolved Oxygen Meter and YSI 5700 Dissolved Oxygen Probe.

Reagents and Apparatus:

1. YSI 54ARC Dissolved Oxygen Meter
2. YSI 5720A B.O.D. Bottle Probe
3. Membrane and KCl kit, standard, YSI 5775
4. Replacement "O" ring, YSI Part #5945
5. Beater boot assembly, YSI Part #5486

Operating Procedure:

I. Preparing the Probe

All YSI 5700 Series Probes have similar sensors and should be cared for in the same manner. They are precision devices relying on good treatment if high accuracy measurements are to be made. Prepare the probe as follows.

ALL PROBES ARE SHIPPED DRY - FOLLOW THESE INSTRUCTIONS
TO PREPARE FOR USE

1. Prepare the electrolyte by dissolving the KCl crystals in a dropper bottle with Milli-Q water. Fill the bottle to the top.
2. Remove the "O" ring and membrane. Thoroughly rinse the sensor with KCl solution.
3. Fill the probe with electrolyte as follows (see Figure 1):
 - a. Grasp the probe with your left hand.
 - b. Fill the sensor body until no more air bubbles appear. Tap the probe against the countertop to dislodge any air bubbles adhering to the sensor.
 - c. Secure a membrane under your left thumb. Add more electrolyte to the probe until a large meniscus completely covers the gold cathode. NOTE: Handle membrane material with care, keeping it clean and dust free, touching it only at the ends.
 - d. With the thumb and forefinger of your other hand, grasp the free end of the membrane.

- e. Using a continuous motion stretch the membrane UP, OVER, and DOWN the other side of the sensor. Stretching forms the membrane to the contour of the probe. The membrane can be stretched to approximately 1 1/2 times its normal size.
 - f. Secure the end of the membrane under the forefinger of the hand holding the probe.
 - g. Roll the "O" ring over the end of the probe. There should be no wrinkles in the membrane or trapped air bubbles. Some wrinkles may be removed by lightly tugging on the edges of the membrane beyond the "O" ring.
 - h. Trim off excess membrane with scissors or sharp knife. Check that the stainless steel temperature sensor is not covered by excess membrane.
4. Shake off excess KCl.
 5. Store the probe in a 800 bottle containing about 1 inch of water.
 6. Membranes average replacement is 2-4 weeks. If the electrolyte in the probe is allowed to evaporate, air bubbles form under the membrane. If air bubbles are noted under the membrane or if the membrane becomes damaged, thoroughly flush the reservoir with fresh KCl and install a new membrane as described above.
 7. Replace the membrane if erratic readings are observed or if calibration is not stable.

NOTE: The gold cathode should always be bright and untarnished. If it is tarnished (which can result from contact with certain gases) or plated with silver (which can result from extended use with a loose or wrinkled membrane), return it to the factory for service. Never use chemicals or abrasives in an attempt to clean it.

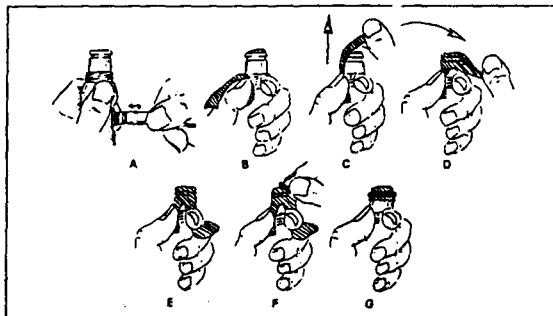


Figure 1

II. Preparing the YSI Instrument

It is important that the instrument be placed in the intended operating position; vertical, tilted, or on its back - before it is prepared for use and calibrated. (See Figure 8). Recalibration may be necessary when the instrument operating position is changed. After preparing the probe proceed as follows:

1. With switch in the OFF position, adjust the meter pointer to Zero with the screw in the center of the meter panel. Readjustment may be necessary if the instrument operating position is changed.
2. Switch to RED LINE and adjust with the RED LINE knob until the meter needle aligns with the red mark at the 31°C position.
3. Switch to ZERO and adjust to zero with zero control knob.
4. Attach the prepared probe to the PROBE connector of the instrument and adjust the retaining ring finger tight.
5. For optimum probe stabilization, let the meter and probe equilibrate for 15 minutes before calibrating.

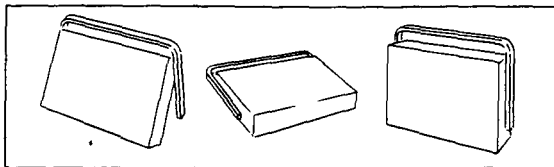


Figure 2

III. Calibration

The operator has a choice of three calibration methods: Winkler titration, Saturated Water, and Air. The three methods are described in the following paragraphs. The Winkler titration is the preferred method of calibration.

Winkler Titration

1. Determine the dissolved oxygen in two samples from the aerated water source using the Winkler titration technique (refer to the Dissolved Oxygen SOP) and average the values. If the values differ by more than 0.5 mg/L, discard the samples and repeat.

2. Place the YSI probe in the third sample and stir.
3. Switch to desired mg/L scale range and adjust with the CALIBRATION control to the average value determined in Step 1. Allow the probe to remain in the sample for at least two minutes before setting the calibration value, and leave in the sample for an additional 2 minutes to verify stability (Readjust if necessary).

Saturated Water Calibration

1. Air saturate a volume of water by aerating for at least 1 hour at a constant temperature (preferably room temperature).
2. Place the probe in the sample and stir. Switch to TEMPERATURE scale. Refer to Calibration Table I for the mg/L value corresponding to the temperature.
3. Determine local altitude or the "true" atmospheric pressure (note that "true" atmospheric pressure is as read on a barometer. Weather Bureau reporting of atmospheric pressure is corrected to sea level). Using Calibration Table II determine the correction factor for your pressure or altitude.
4. Multiply the mg/L value from Table I by the correction factor from Table II to determine the corrected calibration value for your conditions.

EXAMPLE: Assume temperature = 21°C and altitude = 1000 feet. From Table I the calibration value for 21°C is 8.9 mg/L. From Table II the correction factor for 1000 feet is about 0.96. The corrected calibration value is $8.9 \text{ mg/L} \times 0.96 = 8.54 \text{ mg/L}$.

5. Switch to an appropriate mg/L scale range and adjust the CALIBRATE knob while stirring until the meter reads the corrected calibration value from Step 4. Leave the probe in the sample for two minutes to verify calibration stability. Readjust if necessary.

Air Calibration - Fresh Water

1. Place the probe in a 800 bottle partially filled with water. Wait approximately 10 minutes for temperature stabilization. This may be done simultaneously while the probe is stabilizing.
2. Switch to TEMPERATURE and read. Refer to Table I - Solubility of Oxygen in Fresh Water, and determine calibration value.
3. Determine altitude or atmospheric correction factor using Table II.

4. Multiply the calibration value from Table I by the correction factor from Table II.

EXAMPLE: Assume temperature = 21°C and altitude = 1000 feet. From Table I the calibration value for 21°C is 8.9 mg/L. From Table II the correction factor for 1000 feet is about 0.96. Therefore the corrected calibration value is $8.9 \text{ mg/L} \times 0.96 = 8.54 \text{ mg/L}$.

5. Switch to an appropriate mg/L range and adjust the CALIBRATE knob until the meter reads the corrected calibration value from Step 4. Wait two minutes to verify calibration stability. Readjust if necessary.

IV. Dissolved Oxygen Measurement

1. With the instrument prepared for use and the probe calibrated, place the probe in the sample to be measured and turn on stirring boot.
2. Allow sufficient time for probe to stabilize to sample.
3. Read dissolved oxygen directly from scale.

V. Maintenance of the Stirrer Boot

1. The probe uses a flexible stirring boot to transmit motion from the sealed motor housing to the sample. If the boot shows signs of cracking or other damage which may allow leaking into the motor housing, the boot must be replaced.
2. In fresh water applications boot life is normally several years, but this may be shortened by exposure to hydrocarbons, moderate to strong acids or bases, ozone, or direct sunlight. For maximum life rinse the boot with deionized water after use in contaminated samples.
3. Boot replacement is as follows:
 - a. Pull off old assembly and clean shaft.
 - b. Slide on new assembly making sure the back spring is on the grooved area of the shaft. A small amount of rubber cement may be used.
 - c. Check that there is sufficient clearance between the tip and the end of the shaft to permit turning without binding.

TABLE I
Solubility of Oxygen in Fresh Water

Temperature °C	mg/L Dissolved Oxygen	Temperature °C	mg/L Dissolved Oxygen
0	14.60	23	8.56
1	14.19	24	8.40
2	13.81	25	8.24
3	13.44	26	8.09
4	13.09	27	7.95
5	12.75	28	7.81
6	12.43	29	7.67
7	12.12	30	7.54
8	11.83	31	7.41
9	11.55	32	7.28
10	11.27	33	7.16
11	11.01	34	7.05
12	10.76	35	6.93
13	10.52	36	6.82
14	10.29	37	6.71
15	10.07	38	6.61
16	9.85	39	6.51
17	9.65	40	6.41
18	9.45	41	6.31
19	9.26	42	6.22
20	9.07	43	6.13
21	8.90	44	6.04
22	8.72	45	5.95

Source: Derived from 16th Edition "Standard Methods for the Examination of Water and Wastewater".

This table shows the amount of oxygen in mg/L that is dissolved in air saturated fresh water at sea level (760 mm Hg atmospheric pressure) as temperature varies from 0° to 45°C.

Table II
Correction for Atmospheric Pressure

Atmospheric Pressure mm/Hg	or	Equivalent Altitude Ft.	=	Correction Factor
775		540		1.02
760		0		1.00
745		542		.98
730		1094		.96
714		1688		.94
699		2274		.92
684		2864		.90
669		3466		.88
654		4082		.86
638		4756		.84
623		5403		.82
608		6065		.80
593		6744		.78
578		7440		.76
562		8204		.74
547		8939		.72
532		9694		.70
517		10472		.68
502		11273		.66

Source: Derived from 16th Edition "Standard Methods for the Examination of Water and Wastewater".

This table shows the correction factor that should be used to correct calibration value for the effects of atmospheric pressure or altitude. Find true atmospheric pressure in the left hand column and read across to the right hand column to determine the correction factor. (Note that "true" atmospheric pressure is as read on a barometer. Weather Bureau reporting of atmospheric pressure is corrected to sea level.) If atmospheric pressure is unknown, the local altitude may be substituted. Select the altitude in the center column and read across to the right hand column for the correction factor.

YSI 5700 SERIES DISSOLVED OXYGEN PROBES INSTRUCTIONS

The probes described in these instructions are designed for direct use with YSI Models 50, 51B, 54ABP, 54ARC, 56, 57 and 58 Dissolved Oxygen Meters. The probes can also be used with discontinued YSI Models 51A, 54BP and 54RC Dissolved Oxygen Meters when the YSI 5735 Cable Adapter is employed.

PRINCIPLES OF OPERATION

YSI 5700 Series Probes are polarographic sensors. A thin permeable membrane stretched over the sensor isolates the electrodes from the environment, but allows gases to enter. When a polarizing voltage is applied across the sensor, oxygen that has passed through the membrane reacts at the cathode, causing a current to flow.

The membrane passes oxygen at a rate proportional to the difference across it in partial pressure of oxygen. Since oxygen is rapidly consumed at the cathode, it can be assumed that the oxygen pressure under the membrane is zero. Hence, the force causing the oxygen to diffuse through the membrane is proportional to the partial pressure of oxygen outside the membrane. As the oxygen partial pressure varies, both the oxygen diffusion through the membrane and the probe current will change proportionally.

SPECIFICATIONS

Cathode: Gold

Anode: Silver

Membrane: .001" FEP Teflon, standard

Electrolyte: Half saturated KCl

Temperature Range: -5° to 45°C

15° to 35°C for the 5760 probe

Temperature Accuracy: $\pm 0.2^\circ\text{C}$

Temperature Compensation: (see instrument specifications)

Polarizing Voltage: 0.8 Volts (nominal)

Probe Current in Air at 30°C: 19 microamps (nominal)

in Nitrogen at 30°C: 0.15 microamps or less

Response Time: Typical response for dissolved oxygen, using standard membranes, is 90% in 10 seconds at a constant temperature of 30°C.

Response at low dissolved oxygen levels is typically 90% in 30 seconds.

ACCESSORIES AND REPLACEMENT PARTS

YSI 5492A Battery Pack for Models 51B and 54A (Powers the submersible stirrers.)

YSI 5735 Cable Adapter (Mates 5700 Series probes with discontinued YSI Models 51A, 54BP and 54RC Dissolved Oxygen Meters)

Accessories for the 5720A, 5739 and 5750

YSI 5680 Probe Reconditioning Kit. Includes a sanding tool and ten adhesive disks.

YSI 5775 Membrane and KCl Kit, Standard. Includes two 15-membrane packets (.001" thick standard FEP Teflon membranes) and a 30 ml bottle of KCl with Kodak Photo Flo.

YSI 5776 Membrane and KCl Kit, High Sensitivity. Includes two 15-membrane packets (.0005" thick FEP Teflon membranes) and a 30 ml bottle of KCl with Kodak Photo Flo. Used for measurements below 15°C and/or for low oxygen levels

YSI 5793 .001" membranes, 10-membrane packet

YSI 5794 .0005" membranes, 10-membrane packet

YSI 5945 O-ring pack (Contains replacement sensor O-rings)

Accessories for the 5720A Only

YSI 5486 Stirrer Boot Assembly

Accessories for the 5739 Only

YSI 5075A Calibration Chamber

YSI 5986 Diaphragm Kit

YSI 5740-10 detachable 10' cable

YSI 5740-25 detachable 25' cable

YSI 5740-50 detachable 50' cable

YSI 5740-100 detachable 100' cable

YSI 5740-150 detachable 150' cable

YSI 5740-200 detachable 200' cable

YSI 5791A Submersible Stirrer with 50' cable for stirrer only

YSI 5795A Submersible Stirrer with 50' combined probe and stirrer cable

YSI 5720A BOD BOTTLE PROBE

The 5720A bottle probe (Figure 1) is used for measuring dissolved oxygen in standard BOD bottles. It is provided with a stirrer powered by a DC supply available for 115 or 230 VAC input.

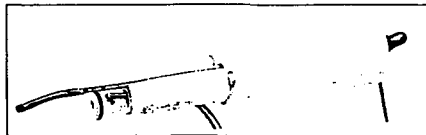


Figure 1. The YSI 5720A Probe



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To use the 5720A, plug the stirrer power supply into line power and the probe plug in the instrument. With the stirrer off, place the tapered probe end into a filled the BOD bottle and turn on the stirrer. The probe should be operated with a minimum of trapped air in the bottle. A slight amount of air in the unstirred region at the top may be neglected, but no bubble should be permitted around the sensor. CAUTION: The motor housing is not waterproof; do not submerge this probe beyond the part that is inserted into a BOD bottle.

Stirrer Boot (YSI 5486)

The 5720A uses a flexible stirring boot to transmit motion from the motor housing to the sample. If the boot shows signs of cracking or other damage liable to allow leakage into the motor housing, it must be replaced. Running the 5720A with a damaged stirring boot could cause permanent motor damage. Boot life may be shortened by exposure to hydrocarbons, moderate to strong acids or bases, ozone, or direct sunlight. For maximum life, rinse the boot after each use. Boots are replaced as follows:

1. Pull off the old assembly and clean the stir rod housing.
2. Slide on the new assembly, making sure the back spring is over the grooved area of the stir rod housing. A drop of alcohol will aid installation by providing lubrication.
3. Do not permit the stir rod to press against the end of the stirrer boot tip or it will bind.

YSI 5739 DISSOLVED OXYGEN PROBE

The 5739 probe system consists of the probe body plus a detachable cable (see Figure 2). The detachable cable is a convenience feature that facilitates changing cable lengths and replacing damaged cables or probes. The probe and cable assembly is held together with a threaded retainer. The assembly is not intended for casual disconnection; cable and probe should be separated only when necessary.

To detach the cable, unscrew the retainer and slide it down the cable to expose the connector. Pull gently on the connector until it comes away from the probe body. If the O-ring is frayed or damaged, replace it: a replacement O-ring is supplied with each 5740 cable. Reassemble by pushing the connector into the probe body, rotating it until the two halves mate. A light coating of silicone grease on the O-ring will make reassembly easier. Be sure the connector is dry; otherwise, erratic readings may result. Screw on the retainer finger-tight only.

Pressure Compensation

The 5739 probe has a unique pressure compensating system that helps assure accurate readings at great depths. Pressure compensation is effective to 1/2% of reading with pressures up to 100 psi (230 feet of water). The compensating system does not normally require service and should not be taken apart. However, if electrolyte is leaking through the diaphragm, or if there is an obvious puncture, the diaphragm must be replaced. A spare is supplied with the probe. Use a coin to unscrew the retaining plug and remove the washer and diaphragm. With distilled water, flush any salt crystals from the reservoir, install a new diaphragm (flat side out), replace the washer and securely screw in the retaining plug.

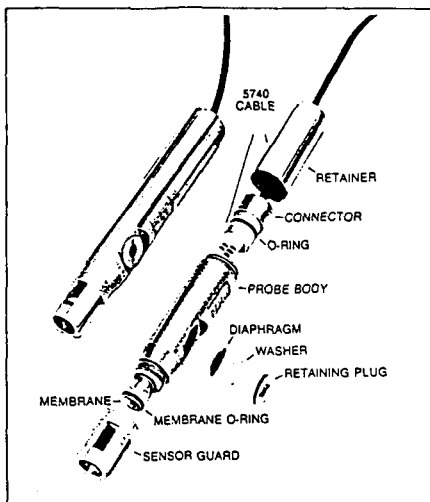


Figure 2. The YSI 5739 Probe

YSI 5750 BOD BOTTLE PROBE

The 5750 (Figure 3) is similar to the 5720A except that it does not have a stirrer. Agitation of the sample must be provided by other means, such as a magnetic stirrer.

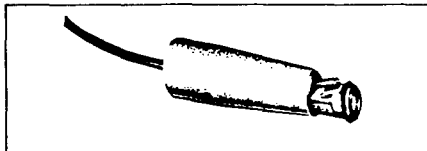


Figure 3. The YSI 5750 Probe

PROBE PREPARATION

All probes are shipped dry. You must follow these instructions when preparing a new probe or when changing membranes. Prepare the electrolyte by dissolving the KCl crystals which are supplied in a dropper bottle that should be filled to the neck with distilled water and shaken until the crystals are dissolved.

1. Unscrew the sensor guard (5739 only). Remove the O-ring and membrane, then thoroughly rinse the sensor with distilled water.
2. To fill the probe with electrolyte and install a new membrane, follow these steps:
 - a. Grasp the probe in your left hand. (See the sketches in Figure 4.) When preparing the 5739 probe, the pressure compensating port should be to the right. Successively fill the sensor body with electrolyte while pumping the diaphragm with the eraser end of a pencil or a similar soft, blunt tool. Continue filling and pumping until no more

air bubbles appear. For ease in preparing the 5720A, the stirring rod should be to the left. When preparing the 5720A or 5750 probes, simply fill the sensor body until no more air bubbles appear.

- b. Secure a membrane between your left thumb and the probe body. Add more electrolyte to the probe until a large meniscus completely covers the gold cathode. NOTE: Handle membrane material with care, touching it at the ends only.
 - c. With the thumb and forefinger of your other hand, grasp the free end of the membrane.
 - d. With a continuous motion, stretch it up, over and down the other side of the sensor. Stretching forms the membrane to the contour of the probe.
 - e. Secure the end of the membrane under the forefinger of your left hand while holding the probe.
 - f. Roll the O-ring over the end of the probe, being careful not to touch the membrane surface. For the 5720A, start at the right side of the sensor and roll the O-ring toward the stirring rod. There should be no wrinkles in the membrane or trapped air bubbles. Some wrinkles may be removed by lightly tugging on the edges of the membrane beyond the O-ring.
 - g. Trim off excess membrane with scissors or sharp knife. Check that the stainless steel temperature sensor is not covered by excess membrane.
3. Shake off excess KCl. On the 5739, reinstall the sensor guard.

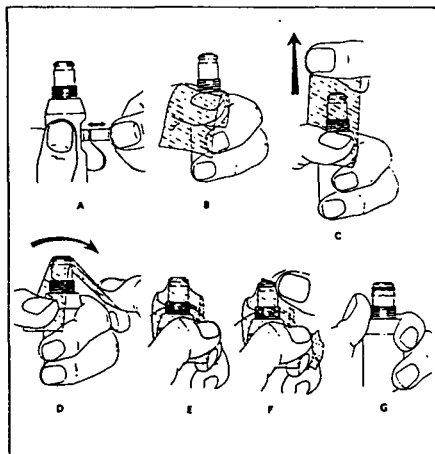


Figure 4. Membrane Application

Probe Storage

A bottomless plastic bottle is provided with the YSI 5739 probe for convenient storage. Place a small piece of moist towel or sponge in the bottle and insert the probe into the open end. This keeps the electrolyte from drying out. The 5720A and 5750 probes can be stored in a BOD bottle containing at least 1" of water.

OPERATING PRECAUTIONS, ALL PROBES

1. Membrane life depends on use. Membranes will last a long time if installed properly and treated with care during use. Erratic readings result from loose, wrinkled or fouled membranes, or from large bubbles in the electrolyte reservoir. If erratic readings, or evidence of membrane damage occur, you should replace the membrane and KCl. The average replacement interval is two to four weeks; electrolyte in constant or heavy use will be exhausted in about two weeks.

If the sensor O-ring on any probe is worn or loose, replace it with the O-ring provided in the YSI 5945 O-ring Pack.

2. The gold cathode should always be bright and untarnished. If it is tarnished (which can result from contact with certain gases) or plated with silver (which can result from extended use with a loose or wrinkled membrane), it needs to have its surface restored. Probes may either be returned to the factory, or cleaned with the YSI 5680 Probe Reconditioning Kit; never use chemicals or any abrasive not supplied with this kit.

3. It is also possible that the silver anode may become contaminated, which will prevent successful calibration. Try soaking the probe overnight in a 3% ammonia solution; rinse with deionized water, recharge with electrolyte, and install a new membrane. If still unable to calibrate after several hours, return the probe for service.

4. Hydrogen sulfide, sulfur dioxide, halogens, and neon are interfering gases. If you suspect erroneous readings, it may be necessary to determine if these are the cause.

These gases have been tested for response:		
100% Carbon Monoxide	less than 1%	
100% Carbon Dioxide	around 1%	
100% Hydrogen	less than 1%	
100% Chlorine	2/3 O ₂ response	
100% Helium	none	
100% Nitrous Oxide	1/3 O ₂ response	
100% Ethylene	none	
100% Nitric Oxide	1/3 O ₂ response	

5. The correct liquid level in BOD bottles is achieved by overfilling, then inserting a stopper and pouring off the excess. When using a YSI 5760 or a 5720A probe in a filled BOD bottle, be careful to insert it slowly to avoid sample overflow.

6. When using the 5720A in samples containing heavy particulate solids, additional stirring may be needed. Inverting the stoppered bottle immediately before use will usually provide adequate mixing.

CALIBRATION

Daily calibration is generally appropriate. Calibration can be disturbed by physical shock, touching the membrane, fouling of the membrane or drying out of the electrolyte. Check calibration after each series of measurements, and in time you will develop a realistic schedule for recalibration. When probes are not in use, store them as recommended in Probe Preparation.

Probes may be calibrated by Winkler Titration or by the Water Saturated Air method. Experience has shown that air calibration is quite reliable, yet far simpler than titration. Both methods are described here. Consult the manual for your particular instrument for more complete instructions.

Winkler Titration

1. Draw a volume of water from a single source and carefully divide it into four samples. Determine the oxygen in three of the samples using the Winkler Titration technique and average the three values. If one of the values differs from the other two by more than 0.5 mg/L, discard it and average the two values remaining.

2. Using the probe-meter system you are calibrating, place the probe into the fourth sample and stir.

3. Switch to the desired mg/L range and adjust the CALIBRATION control to the average value determined in step 1. Allow the probe to remain in the sample for at least 5 minutes before setting the calibration value, then leave it in the sample for an additional two minutes to verify stability. Readjust if necessary.

Air Calibration

1. Place the probe in a BOD bottle containing about 1 inch of water. Wait approximately ten minutes for temperature stabilization.

The 5739 probe can be placed in the YSI 5075A Calibration Chamber or in the small calibration bottle supplied with the probe (the one with the hole in the bottom) along with a few drops of water, or a moistened towel or cloth.

2. Read the temperature and refer to the instrument Calibration Table to determine the calibration value.

NOTE: To achieve the stated accuracy of measurement, the probe must be stabilized before calibrating. The calibration temperature should be within 5 degrees of the sample temperature.

3. Determine the atmospheric correction factor (see Instrument instructions).

4. Multiply the calibration value by the correction factor.

5. Switch your instrument to an appropriate mg/L range and adjust the CALIBRATE control until the meter reads the corrected calibration value from step 4. Without changing the calibration setup, monitor the readings for an additional 3 minutes to verify calibration stability. Readjust if necessary.

WARRANTY AND REPAIR

All YSI products carry a one-year warranty on workmanship and parts, exclusive of batteries. Damage through accident, misuse, or tampering will be repaired at a nominal charge, if possible, when the item is returned to the factory or to an authorized YSI Dealer. Electrode cleaning is not covered by warranty.

If you are experiencing difficulty with any YSI product, it may be returned for repair, even if the warranty has expired. YSI maintains complete facilities for prompt servicing on all its products. This warranty is limited to repair or replacement (YSI's option) at no charge.

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Memo to J:11

YSI Inc.
attn: Product Service
Billing address
name P.O. #4-
& ph no. order 45387

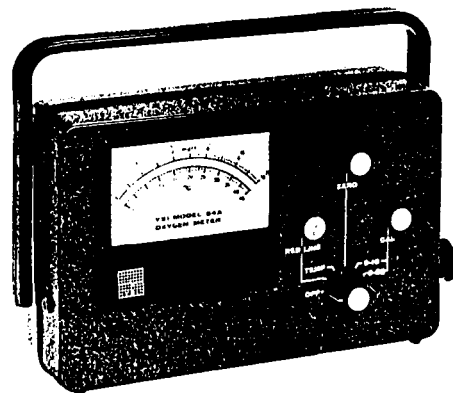


YSI Incorporated

Yellow Springs Instrument Co., Inc., Yellow Springs, Ohio 45387 USA • Phone 513 767-7241 • NIK 513-411ELP • Fax 513 767-9353 • Telex 205437

Item 004483 Part A05730 N November 1988

**INSTRUCTION MANUAL
YSI MODELS 54ARC AND 54ABP
DISSOLVED OXYGEN METERS**



Scientific Division
Yellow Springs Instrument Co., Inc.
Yellow Springs, Ohio 45387, U.S.A. • Phone 513-767-7241

SUMMARY OF OPERATING INSTRUCTIONS

1. CALIBRATION

- A. Switch instrument to OFF and adjust meter mechanical zero.
- B. Switch to RED LINE and adjust.
- C. Prepare probe for operation, connect to instrument, wait up to 15 minutes for probe to stabilize. Probe can be in calibration chamber or ambient air.
- D. Switch to ZERO and adjust to "0" on mg/l scale.
- E. Switch to TEMP and read on °C scale.
- F. Use probe temperature and true local atmospheric pressure (or feet above sea level) to determine calibration values from Tables I and II. (See pages 14 and 15).
EXAMPLE: Probe temperature = 21°C; Altitude = 1000 feet. From Table I the calibration value for 21°C is 8.9 mg/l. From Table II the altitude factor for 1000 feet is approximately .96. The correct calibration value, then, is:

$$8.9 \text{ mg/l} \times .96 \text{ factor} = 8.54 \text{ mg/l}$$

- G. Switch to 0-10 or 0-20 mg/l range and adjust meter with CAL control to calibration value determined in Step F.
NOTE: It is desirable to calibrate probe in a high humidity environment. (See calibration section for more detail).

2. MEASUREMENT

- A. Place probe in sample and stir.
- B. Allow sufficient time for probe to stabilize to sample temperature and dissolved oxygen.
- C. Read dissolved oxygen on appropriate range (1-10 or 0-20 mg/l)
- D. We recommend the instrument be left on between measurements to avoid the necessity to repolarize the probe.

3. GENERAL CARE

- A. Recharge batteries in the YSI Model 54ARC when the instrument can no longer be red lined. Recharge 16-20 hours. Replace with Burgess CD-6 or equivalent. Replace batteries in the YSI Model 54ABP when red line cannot be set with Panasonic UM-2N or equivalent.
- B. Membranes will last indefinitely, depending on usage. Average replacement is 2-4 weeks. Probe should be stored in humid environment to prevent drying out.
- C. Calibrate daily.

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GENERAL DESCRIPTION

The YSI Models 54ARC and 54ABP Dissolved Oxygen Meters are intended for dissolved oxygen and temperature measurement in water and wastewater applications, but are also suitable for use in certain other liquids. Dissolved Oxygen is indicated in mg/l (milligrams per liter) on 0-10 and 0-20 mg/l scales. Temperature is indicated in °C on a -5° to +45°C scale. Both dissolved oxygen ranges are automatically temperature compensated for solubility of oxygen in water and permeability of the probe membrane.

The probes use Clark-type membrane covered polarographic sensors with built-in thermistors for temperature measurement and compensation. A thin, permeable membrane stretched over the sensor isolates the sensor elements from the environment, but allows oxygen and certain other gases to enter. When a polarizing voltage is applied across the sensor, oxygen that has passed through the membrane reacts at the cathode, causing a current to flow.

The membrane passes oxygen at a rate proportional to the pressure difference across it. Since oxygen is rapidly consumed at the cathode, it can be assumed that the oxygen pressure inside the membrane is zero. Hence, the force causing the oxygen to diffuse through the membrane is proportional to the absolute pressure of oxygen outside the membrane. If the oxygen pressure increases, more oxygen diffuses through the membrane and more current flows through the sensor. A lower pressure results in less current.

Power to operate the system is provided by internal batteries in the instruments, rechargeable batteries in the YSI Model 54ARC and disposable batteries in the YSI Model 54ABP.

SPECIFICATIONS

I. Instrument

Oxygen Measurement

Ranges: 0-10 and 0-20 mg/l (0-5 and 0-10 mg/l with YSI 5776 High Sensitivity Membrane)

Accuracy: $\pm 1\%$ of full scale at calibration temperature (± 0.1 mg/l and 0-10 scale)

Readability: .05 mg/l on 0-10 scale; 0.1 mg/l on 0-20 scale.

Temperature Measurement

Ranges: -5° to +45°C

Accuracy: $\pm 0.7^\circ\text{C}$, including probe

Readability: 0.25°C

Temperature Compensation

$\pm 1\%$ of D.O. reading for measurements made within $\pm 5^\circ\text{C}$ of calibration temperature.

$\pm 3\%$ of D.O. reading over entire range of -5 to +45°C Probe temperature.

System Response Time

Typical response for temperature and D.O. readings is 90% in 10 seconds at constant temperature of 30°C with YSI 5775 Membranes. D.O. response at low temperature and low D.O. is typically 90% in 30 seconds. YSI 5776 High Sensitivity Membranes can be used to improve response at

low temperature and low D.O. concentrations. If response time under any operating conditions exceeds two minutes, probe service is indicated.

Operating Temperature Range

Instrument and probe operating range is -2° to +45°C. Large ambient temperature changes will result in 2% loss of accuracy unless Red Line and Zero are reset.

Recorder Output

0 to 114-136 mV. Recorder should have 50,000 ohms minimum input impedance.

Power Supply

YSI Model 54ABP: (4) 1.5 volt carbon zinc batteries provide approximately 1000 hours operation. Replace with Panasonic UM-2N or equal.

YSI Model 54ARC: (4) 1.25 volt Ni-Cad rechargeable cells (Burgess CD-6 or equal) provide approximately 100 hours of operation between charges.

II. Probe

Cathode: Gold

Anode: Silver

Membrane: .001" FEP Teflon (.0005" FEP Teflon available)

Electrolyte: Half Saturated KCl

Temperature Compensation: (See SPECIFICATIONS, I. Instrument)

Pressure Compensation: Effective 1/2% of reading to pressures of 100 psi (230 ft. water)

Polarizing Voltage: 0.8 volts nominal

Probe Current: Air at 30°C = 19 microamps nominal

Nitrogen at 30°C = .15 microamps or less

III. Accessories and Replacement Parts

YSI 5720A — Self Stirring B.O.D. Bottle Probe

YSI 5750 — Non Stirring B.O.D. Bottle Probe

YSI 5739 — Oxygen Temperature Probe for field use. Combine with one of the following cables for desired lead length.

YSI 5401 — Battery Charger Eliminator 115V

YSI 5402 — Battery Charger Eliminator 230V

Detachable leads for use with YSI 5739

YSI 5740-10	10' cable
YSI 5740-25	25' cable
YSI 5740-50	50' cable
YSI 5740-100	100' cable
YSI 5740-150	150' cable
YSI 5740-200	200' cable

YSI 5492A — Battery Pack Operates YSI 5791A and 5795A Submersible Stirrers

- YSI 5791A — Submersible Stirrer for field use
- YSI 5795A — Submersible Stirrer for field use
- YSI 5075A — Calibration Chamber for use with field probe
- YSI 5890 — Carrying Case
- YSI 5775 — Membrane and KCl Kit, Standard — includes 2 each 15-membrane packets (.001" thick standard membranes) and a 30 ml bottle KCl with Kodak Photo Flo.
- YSI 5776 — Membrane and KCl Kit, High Sensitivity — includes 2 each 15-membrane packets (.0005" thick membranes) and a 30 ml bottle KCl with Kodak Photo Flo.
- YSI 5945 — "O" Ring Pack — includes (6) "O" rings for each YSI D.O. Probe.
- YSI 5486 — Beater Boot Kit — includes (1) A-05486 Boot, (1) A-05484 Tip, (2) A-05485 Spring. Used only on 5720A and discontinued 5420A and 5720.
- YSI 5986 — Diaphragm Kit for use only with YSI 5739 D.O. Probe.
- YSI 5734 — Adaptor makes it possible to use discontinued YSI 5400 Series Probes with YSI Models 54ARC and 54ABP.
- YSI 5735 — Adaptor makes it possible to use YSI 5739, 5720A and 5750 Probes with discontinued YSI Models 54RC and 54BP.

OXYGEN PROBES AND EQUIPMENT

There are three oxygen probes for use with the YSI Models 54ARC and 54ABP Dissolved Oxygen Meters. Descriptions of where they are used are contained in the following paragraphs.

I. YSI 5739 D.O. Probe

The YSI 5739 probe, with built-in lead weight and pressure compensation, is an improved design that replaces the discontinued YSI 5418, 5419, 5718 and 5719 probes. (See Figure 1)

For user convenience the probe is equipped with a disconnecting cable to facilitate changing cable lengths and replacing damaged cables or probes. The probe and cable assembly is held together with a threaded retaining nut. The connection is *not* designed for casual disconnection and should only be disconnected when necessary.

To disconnect the cable unscrew the retaining nut and slide it down the cable to expose the connector. Pull gently on the cable and connector until the connector comes away from the probe body.

To reassemble, inspect the connector and "O" ring for cleanliness. If the "O" ring is frayed or damaged remove it by squeezing it in the groove causing it to bulge, then roll it out of the groove and off the connector. A replacement "O" ring is supplied with the cable.

Push the connector into the probe body, rotating it until the two halves mate. A light coating of vaseline or silicone grease on the "O" ring will make reassembly easier. Air trapped between the connector halves which may cause them to spring apart slightly, is normal. Screw on the retaining nut, *hand tight only*. NOTE: If erratic readings are experienced, disconnect the cable and inspect for water. If present, dry out and reconnect, replacing the "O" ring, if necessary.

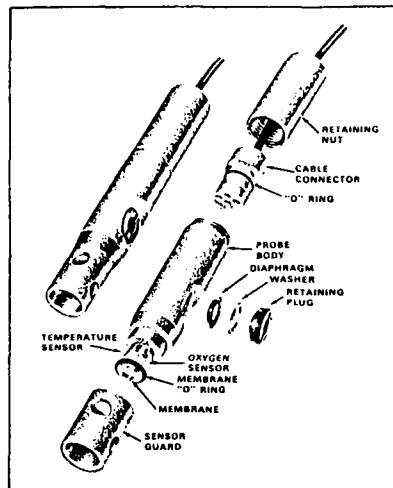


Figure 1

Pressure Compensation

The vent on the side of the probe is part of a unique pressure compensating system that helps assure accurate readings at great depths of water. Pressure compensation is effective to 1/2% of reading with pressures to 100 psi (230 ft. water). The quantity of air bubbles trapped under the membrane determines how serious the pressure error will be, which is why proper preparation of the probe is essential. (See OPERATING PROCEDURES.) The system is designed to accommodate a small amount of trapped air and still function properly, but the amount should be kept to a minimum.

The compensating system normally does not require servicing and should not be taken apart. However, if electrolyte is leaking through the diaphragm or if there is an obvious puncture, the diaphragm must be replaced. A spare is supplied with the probe. Using a coin unscrew the retaining plug and remove the washer and the diaphragm, flush any salt crystals from the reservoir, install the new diaphragm (convolution side in), replace the washer, and screw in the retaining plug.

II. YSI 5720A B.O.D. Bottle Probe

The YSI 5720A B.O.D. Bottle Probe replaces the discontinued YSI 5420A B.O.D. Bottle Probe for measuring dissolved oxygen and temperature in standard B.O.D. bottles. It is provided with an agitator for stirring the sample solution, available in models for 117VAC (95-135VAC, 50-60 Hz) or 230VAC (190-250VAC, 50-60 Hz) operation. (See Figure 2)

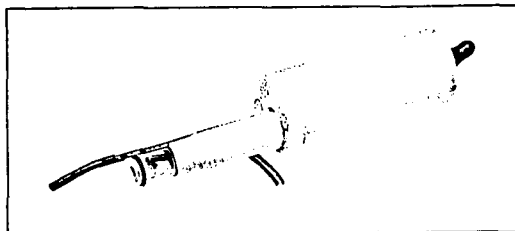


Figure 2

When using the probe, plug the agitator power supply into line power and the probe plug into the instrument. With the agitator turned off place the tapered probe end into the B.O.D. bottle and switch agitator "ON" with switch on top of probe. The probe should be operated with a minimum of trapped air in the B.O.D. bottle. A slight amount of air in the unstirred region at the top of the bottle may be neglected, but no bubbles should be around the thermistor or oxygen sensor.

Stirrer Boot

The probe uses a flexible stirring boot to transmit motion from the sealed motor housing to the sample. If the boot shows signs of cracking or other damage likely to allow leaking into the motor housing, the boot must be replaced.

In fresh water applications boot life is normally several years, but this may be shortened by exposure to hydrocarbons, moderate to strong acids or bases, ozone, or direct sunlight. For maximum life rinse the boot after use in contaminated samples. (See Figure 3)

Boot replacement is as follows:

1. Pull off old assembly and clean shaft.
2. Slide on new assembly making sure the back spring is on the grooved area of the shaft. A small amount of rubber cement may be used.
3. Check that there is sufficient clearance between the tip and the end of the shaft to permit turning without binding.

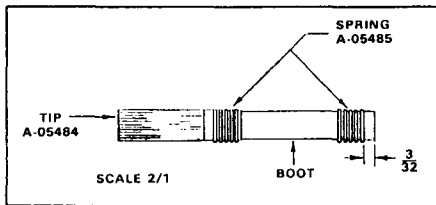


Figure 3



Figure 4

III. YSI 5750 B.O.D. Bottle Probe

The YSI 5750 B.O.D. Bottle Probe replaces the discontinued YSI 5450 B.O.D. Bottle Probe. It is similar to the YSI 5720A B.O.D. Bottle Probe, except that it does not have a stirrer. Agitation of the sample must be provided by other means, such as a magnetic stirrer. (See Figure 4)

IV. Cable Adaptors

All YSI 5700 Series Probes are designed for direct use with the YSI Models 54ARC and 54ABP Dissolved Oxygen Meters. However, to use YSI 5700 probes with the discontinued YSI Models 54RC and 54BP, cable adaptor YSI 5735 is required.

V. YSI 5791A and 5795A Submersible Stirrers

The YSI submersible stirrers are accessories that perform the function of stirring the sample being studied when making dissolved oxygen measurements in the field. The YSI 5791A stirrer can be used with the following dissolved oxygen probes: YSI 5418, 5419, 5718, 5719, and 5739. The YSI 5795A stirrer is only for use with the YSI 5739 Probe. (See Figure 6)

When a stirrer and probe are assembled, the stirrer agitates the sample directly in front of the sensor by means of a rotating eccentric weight which causes the spring-mounted hermetically sealed motor housing to vibrate. An impeller on the end of the motor housing flushes the media across the oxygen sensor. (See sales literature and instruction sheets for further information).

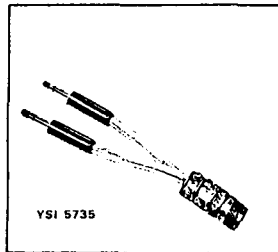


Figure 5

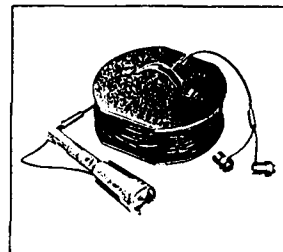


Figure 6

VI. YSI 5492A Battery Pack

The YSI 5492A Battery Pack is designed to attach to the case of all YSI Model 54 Dissolved Oxygen Meters to provide power for operating the submersible stirrers. (See sales literature and instruction sheets for further information).

OPERATING PROCEDURES

I. Preparing the Probe

All YSI 5700 Series Probes have similar sensors and should be cared for in the same manner. They are precision devices relying on good treatment if high accuracy measurements are to be made. Prepare the probes as follows. (See Figure 7)

ALL PROBES ARE SHIPPED DRY — YOU MUST FOLLOW THESE INSTRUCTIONS

1. Prepare the electrolyte by dissolving the KCl crystals in the dropper bottle with distilled water. Fill the bottle to the top.
2. Unscrew the sensor guard from the probe (YSI 5739 only) and then remove the "O" ring and membrane. Thoroughly rinse the sensor with KCl solution.
3. Fill the probe with electrolyte as follows:

A. Grasp the probe in your left hand. When preparing the YSI 5739 probe the pressure compensating vent should be to the right. Successively fill the sensor body with electrolyte while pumping the diaphragm with the eraser end of a pencil or similar soft, blunt tool. Continue filling and pumping until no more air bubbles appear. (With practice you can hold the probe and pump with one hand while filling with the other.) When preparing the YSI 5720A and 5750 probes, simply fill the sensor body until no more air bubbles appear.

B. Secure a membrane under your left thumb. Add more electrolyte to the probe until a large meniscus completely covers the gold cathode. NOTE: Handle membrane material with care, keeping it clean and dust free, touching it only at the ends.

C. With the thumb and forefinger of your other hand, grasp the free end of the membrane.

D. Using a continuous motion *stretch* the membrane *UP, OVER,* and *DOWN* the other side of the sensor. Stretching forms the membrane to the contour of the probe. The membrane can be stretched to approximately 1-1/2 times its normal length.

E. Secure the end of the membrane under the forefinger of the hand holding the probe.

F. Roll the "O" ring over the end of the probe. There should be no wrinkles in the membrane or trapped air bubbles. Some wrinkles may be removed by lightly tugging on the edges of the membrane beyond the "O" ring.

G. Trim off excess membrane with scissors or sharp knife. Check that the stainless steel temperature sensor is not covered by excess membrane.

4. Shake off excess KCl and reinstall the sensor guard.

5. A bottomless plastic bottle is provided with the YSI 5739 probe for convenient storage. Place a small piece of moist towel or sponge in the bottle and insert the probe into the open end. This keeps the electrolyte from dry-

ing out. The YSI 5720A and 5750 probes can be stored in a B.O.D. bottle containing about 1" of water.

6. Membranes will last indefinitely, depending on usage. Average replacement is 2-4 weeks. However, should the electrolyte be allowed to evaporate and an excessive amount of bubbles form under the membrane, or the membrane become damaged, thoroughly flush the reservoir with KCl and install a new membrane.
7. Also replace the membrane if erratic readings are observed or calibration is not stable.
8. "Home brew" electrolyte can be prepared by making a saturated solution of reagent grade KCl and distilled water, and then diluting the solution to half strength with distilled water. Adding two drops of Kodak Photo Flo per 100 ml of solution assures good wetting of the sensor, but is not absolutely essential.
9. The gold cathode should always be bright and untarnished. If it is tarnished (which can result from contact with certain gases) or plated with silver (which can result from extended use with a loose or wrinkled membrane), return it to the factory for service. Never use chemicals or any abrasive.
10. H₂S, SO₂, Halogens, Neon, Nitrous Oxide and CO are interfering gases. If you suspect erroneous readings, it may be necessary to determine if these are the cause. These gases have been tested for response.

100% Carbon Monoxide-Less than 1%	100% Helium-none
100% Carbon Dioxide-Around 1%	100% Nitrous Oxide-1/3 O ₂ response
100% Hydrogen-Less than 1%	100% Ethylene-none
100% Chlorine-2/3 O ₂ response	100% Nitric Oxide-1/3 O ₂ response

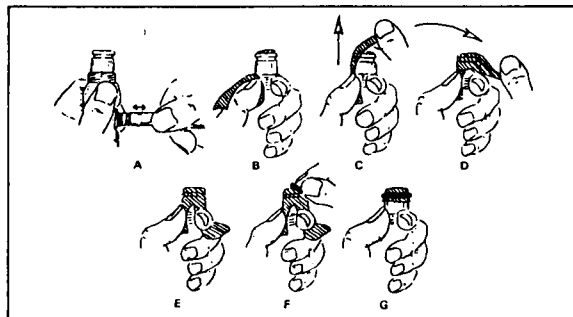


Figure 7

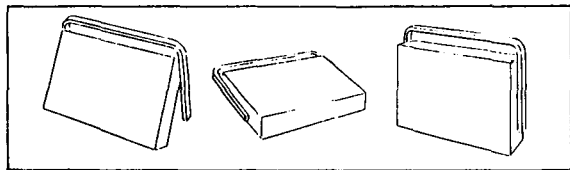


Figure 8

II. Preparing the Instrument

It is important that the instrument be placed in the intended operating position vertical, tilted, or on its back — before it is prepared for use and calibrated. (See Figure 8). Readjustment may be necessary when the instrument operating position is changed. After preparing the probe proceed as follows:

1. With switch in the OFF position, adjust the meter pointer to Zero with the screw in the center of the meter panel. Readjustment may be necessary if the instrument position is changed.
2. Switch to RED LINE and adjust the RED LINE knob until the meter needle aligns with the red mark at the 31°C position.
3. Switch to ZERO and adjust to zero with zero control knob.
4. Attach the prepared probe to the PROBE connector of the instrument and adjust the retaining ring finger tight.
5. Before calibrating allow 15 minutes for optimum probe stabilization. Repolarize whenever the instrument has been OFF or the probe has been disconnected.

III. Calibration

The operator has a choice of three calibration methods — Winkler Titration, Saturated Water, and Air. Experience has shown that air calibration is quite reliable, yet far simpler than the other two methods. The three methods are described in the following paragraphs.

Winkler Titration

1. Draw a volume of water from a common source and carefully divide into four samples. Determine the oxygen in three samples using the Winkler Titration technique and average the three values. If one of the values differs from the other 2 by more than 0.5 mg/l, discard that value and average the remaining two.
2. Place the probe in the fourth sample and stir.
3. Switch to desired mg/l range and adjust the CALIBRATION control to the average value determined in Step 1. Allow the probe to remain in the sample for at least two minutes before setting the calibration value, and leave in the sample for an additional 2 minutes to verify stability. (Readjust if necessary).

Saturated Water

1. Air saturate a volume of water (300-500 cc) by aerating or stirring for at least 15 minutes at a relatively constant temperature.

2. Place the probe in the sample and stir. Switch to TEMPERATURE. Refer to Calibration Table I for the mg/l value corresponding to the temperature.
3. Determine local altitude or the "true" atmospheric pressure (note that "true" atmospheric pressure is as read on a barometer. Weather Bureau reporting of atmospheric pressure is corrected to sea level). Using Calibration Table II determine the correction factor for your pressure or altitude.
4. Multiply the mg/l value from Table I by the correction factor from Table II to determine the corrected calibration value for your conditions.
 EXAMPLE: Assume temperature = 21°C and altitude = 1000 feet. From Table I the calibration value for 21°C is 8.9 mg/l. From Table II the correction factor for 1000 feet is about 0.96. The corrected calibration value is $8.9 \text{ mg/l} \times 0.96 = 8.54 \text{ mg/l}$.
5. Switch to an appropriate mg/l range and adjust the CALIBRATE knob while stirring until the meter reads the corrected calibration value from Step 4. Leave the probe in the sample for two minutes to verify calibration stability. Readjust if necessary.

Air Calibration — Fresh Water

1. Place the probe in moist air. B.O.D. probes can be placed in partially filled (50 ml) B.O.D. bottles. Other probes can be placed in the YSI 5075A Calibration Chamber (refer to the following section describing CALIBRATION CHAMBER) or the small calibration bottle (the one with the hole in the bottom) along with a few drops of water. The probe can also be wrapped loosely in a damp cloth taking care the cloth does not touch the membrane. Wait approximately 10 minutes for temperature stabilization. This may be done simultaneously while the probe is stabilizing.
2. Switch to TEMPERATURE and read. Refer to Table I — Solubility of Oxygen in Fresh Water, and determine calibration value.
3. Determine altitude or atmospheric correction factor using Table II.
4. Multiply the calibration value from Table I by the correction factor from Table II.
 EXAMPLE: Assume temperature = 21°C and altitude = 1000 feet. From Table I the calibration value for 21°C is 8.9 mg/l. From Table II the correction factor for 1000 feet is about 0.96. Therefore, the corrected calibration value is $8.9 \text{ mg/l} \times 0.96 = 8.54 \text{ mg/l}$.
5. Switch to the appropriate mg/l range and adjust the CALIBRATE knob until the meter reads the corrected calibration value from Step 4. Wait two minutes to verify calibration stability.

Readjust if necessary.

Air Calibration — Sea Water

1. Place the probe in moist air. B.O.D. probes can be placed in partially filled (50 ml) B.O.D. bottles. Other probes can be placed in the YSI 5075A Calibration Chamber (refer to the following section describing Calibration Chamber) or the small storage bottle (the one with the hole in the bottom) along with a few drops of water. The probe can also be wrapped loosely in a damp cloth taking care the cloth does not touch the membrane. Wait approximately 10 minutes for temperature stabilization. This may be done simultaneously while the probe is polarizing.

2. Switch to TEMPERATURE and read. Refer to Table III — Solubility of Oxygen in Sea Water, and determine calibration value.
3. Switch to the appropriate mg/l range, and adjust the CALIBRATE knob until the meter reads the calibration value determined in Step 2. Wait 2 minutes to verify calibration stability. Readjust if necessary.

The probe is now calibrated and should hold this calibration value for many measurements. Calibration can be disturbed by physical shock, touching the membrane, or drying out of the electrolyte. Check calibration after each series of measurements and in time you will develop a realistic schedule for recalibration. For best results when not in use, follow the storage procedures recommended for the various probes described under OXYGEN PROBES AND EQUIPMENT. This will reduce drying out and the need to change membranes.

Calibration Chamber

The YSI 5075A Calibration Chamber is an accessory that helps obtain optimum calibration in the field and is also a useful tool for measuring at shallow depths (less than 4').

As shown in Figure (A), it consists of a 4-1/2 foot stainless steel tube (1) attached to the calibration chamber (2), the measuring ring (3), and two stoppers (4) and (5).

For calibration, insert the solid stopper (4) in the bottom of the calibration chamber (2). Push the oxygen probe (6) through the hollow stopper (5) as shown in Figure (B). Place the probe in the measuring ring, Figure (C), and immerse the probe in the sample to be measured for five minutes to thermally equilibrate the probe. Quickly transfer the probe to the calibration chamber (5) draining excess water from the chamber and shaking any excess droplets from

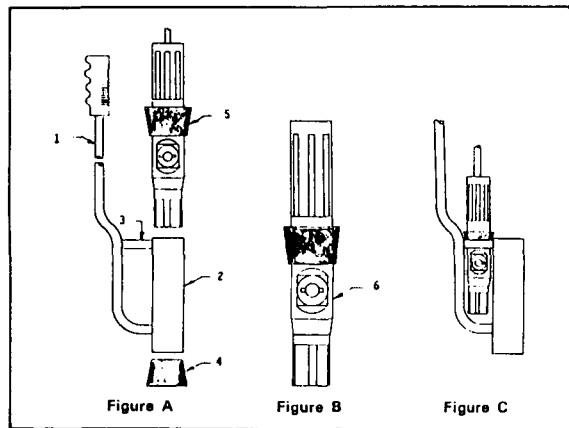


Figure 9

the probe membrane. For maximum accuracy, wet the inside of the calibration chamber with fresh water. This creates a 100% relative humidity environment for calibration. Place the chamber in the sample for an additional five minutes for final thermal equilibrium. Calibrate the probe as described in the air-calibration procedure. Keep the handle above water at all times.

After calibration, return the probe to the measurement ring for shallow measurements. Move the probe up and down, or horizontally, approximately one foot a second while measuring. In rapidly flowing streams (greater than 5/sec-ond) install the probe in the measuring ring with the pressure compensating diaphragm towards the chamber.

IV. Dissolved Oxygen Measurement

With the instrument prepared for use and the probe calibrated, place the probe in the sample to be measured and provide stirring.

1. Stirring for the YSI 5739 Probe can best be accomplished with a YSI submersible stirrer. If the submersible stirrer is not used, provide manual stirring by raising and lowering the probe about 1 ft. per second. If the 5075 Calibration Chamber is used, the entire chamber may be moved up and down in the water at about 1 ft. per second.
2. The YSI 5720A has a built-in power driven stirrer.
3. With the YSI 5750 sample stirring must be accomplished by other means such as with the use of a magnetic stirring bar.
4. Allow sufficient time for probe to stabilize to sample temperature and dissolved oxygen.
5. Read dissolved oxygen.

V. High Sensitivity Membrane

Use of high sensitivity .0005" membranes (YSI 5776) in place of standard .001" membrane (YSI 5775) is recommended when measurements are to be made consistently at low temperatures (less than 15°C). Calibration and readings will be made just as if the standard YSI 5775 Membrane was being used.

The YSI 5776 High Sensitivity Membranes can also be used in certain situations to increase sensitivity at temperatures about 15°C. The ranges thus become 0-5 and 0-10 mg/l. When calibration with high sensitivity membranes is attempted at temperatures greater than 15°C the selector switch must be set to 0-20 mg/l. Multiply the calculated calibration value by 2. For example, at 21°C and 1000 ft. altitude the calibration value would be 8.6×2 or 17.2. Remember the 0-10 and 0-20 mg/l ranges are now 0-5 and 0-10 mg/l, and all mg/l readings must be divided by 2 for a final reading. When operating in this manner accuracy will be degraded slightly.

VI. Recorder Output

Red and black recorder jacks are provided on the YSI Models 54ARC and 54ABP, if you wish to record data while measuring. The high terminal of the recorder is connected to the red tip jack and the low terminal to the black. Output of the YSI 54A at full scale is between 114 to 136 mV.

Use a 50K or higher input impedance recorder and operate it with the terminals ungrounded. The recorder should be operated with its terminals ungrounded. Calibration of the instrument should be checked after connection of the recorder.

Many recorders have an adjustable full scale sensitivity feature. When these recorders are used with the Model 54A, use the 100 millivolt range and adjust the full scale chart deflection when there is full scale meter deflection. Refer to the instruction book for the recorder. For recorders without this feature, a simple divider network as shown below can be constructed. This is adequate to adjust the signal for full scale chart and meter deflection on the 100 mV fixed range recorders.

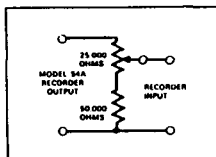


Figure 10

VIII. Calibration Tables

Table I shows the amount of oxygen in mg/l that is dissolved in air saturated fresh water at sea level (760 mmHg atmospheric pressure) as temperature varies from 0° to 45°C.

Table I — Solubility of Oxygen in Fresh Water

Temperature °C	mg/l Dissolved Oxygen	Temperature °C	mg/l Dissolved Oxygen
0	14.60	23	8.56
1	14.19	24	8.40
2	13.81	25	8.24
3	13.44	26	8.09
4	13.09	27	7.95
5	12.75	28	7.81
6	12.43	29	7.67
7	12.12	30	7.54
8	11.83	31	7.41
9	11.55	32	7.28
10	11.27	33	7.16
11	11.01	34	7.05
12	10.76	35	6.93
13	10.52	36	6.82
14	10.29	37	6.71
15	10.07	38	6.61
16	9.85	39	6.51
17	9.65	40	6.41
18	9.45	41	6.31
19	9.26	42	6.22
20	9.07	43	6.13
21	8.90	44	6.04
22	8.72	45	5.95

Source: Derived from 15th Edition "Standard Methods for the Examination of Water and Wastewater."

Table II — Correction for Atmospheric Pressure

Table II shows the correction factor that should be used to correct the calibration value for the effects of atmospheric pressure or altitude. Find true atmospheric pressure in the left hand column and read across to the right hand column to determine the correction factor. (Note that "true" atmospheric pressure is as read on a barometer. Weather Bureau reporting of atmospheric pressure is corrected to sea level.) If atmospheric pressure is unknown, the local altitude may be substituted. Select the altitude in the center column and read across to the right hand column for the correction factor.

Table II

Atmospheric Pressure mmHg	or Equivalent Altitude Ft.	= Correction Factor
775	540	1.02
760	0	1.00
745	542	.98
730	1094	.96
714	1688	.94
699	2274	.92
684	2864	.90
669	3466	.88
654	4082	.86
638	4756	.84
623	5403	.82
608	6065	.80
593	6744	.78
578	7440	.76
562	8204	.74
547	8939	.72
532	9694	.70
517	10472	.68
502	11273	.66

Source: Derived from 15th Edition "Standard Materials for the Examination of Water and Wastewater."

The temperature-solubility relationship of oxygen in sea water is not the same as that in fresh water. For this reason the compensation error when used with sea water is greater than when used with fresh water. For a $\pm 5^{\circ}\text{C}$ span the error could be +2.2% of reading and over the temperature range of -2° to $+30^{\circ}\text{C}$ the error could be 6.3% of reading.

Table III — Solubility of Oxygen in Sea Water

SOLUBILITY OF OXYGEN IN SEA WATER
(Chloride concentration 20,000 mg/l)

Temp. °C	Solubility mg/l	Temp. °C	Solubility mg/l
0	11.41	16	7.91
1	11.11	17	7.78
2	10.83	18	7.61
3	10.56	19	7.47
4	10.30	20	7.33
5	10.05	21	7.20
6	9.82	22	7.07
7	9.59	23	6.95
8	9.37	24	6.83
9	9.16	25	6.71
10	8.96	26	6.60
11	8.77	27	6.49
12	8.58	28	6.38
13	8.41	29	6.28
14	8.24	30	6.18
15	8.07		

Source: Derived from 15th Edition "Standard Materials for the Examination of Water and Wastewater."

Correcting for Salinity

When measuring dissolved oxygen in water samples with a salinity or chlorinity between sea water and fresh water, calibrate the instrument for fresh water and make your measurements. Then correct the data according to the following formula:

FORMULA:

$$A = M \left[1.0 - \left(\frac{Cs/Co [Sf - So]}{Sf} \right) \right]$$

Where: A = Actual DO of sample. (mg/l dissolved O₂)

M = Measured DO with instrument

* Co = Chlorinity of ocean water (20 o/oo Cl ion)

* Cs = Chlorinity of sample (o/oo Cl ion)

Sf = DO of saturated fresh water at 760 mm pressure and at same temperature as sample (mg/l DO, obtain data from charts in instruction manual)

So = DO of saturated ocean water (20,000 mg/l Chloride ion) at 760 mm pressure and at same temperature as sample (mg/l DO, obtain data from instruction manual)

* NOTE: If salinity is used instead of chlorinity the ratio Cs/Co is computed using 36.11 o/oo for Co (salinity of ocean water), and the salinity of your sample of Cs.

EXAMPLE: Measured Data

DO = 4.1

Temp = 22°C

Salinity = 31 o/oo salinity

M = 4.1 mg/l DO from data

Co = 36.11 o/oo salinity from manual

Cs = 31.0 o/oo salinity from data

Sf = 8.8 mg/l DO from Table I in manual

So = 7.1 mg/l DO from Table II in manual

$$\begin{aligned}
 A &= 4.1 \left[1.0 - \left(\frac{[31.0/36.11] [8.8 - 7.1]}{8.8} \right) \right] \\
 &= 4.1 \left[1.0 - \left(\frac{[.86] [1.7]}{8.8} \right) \right] \\
 &= 4.1 \left[1.0 - \left(\frac{1.46}{8.8} \right) \right] \\
 &= 4.1 \left[1.0 - 0.166 \right] \\
 &= 4.1 [0.834] \\
 &= 3.41 \text{ mg/l}
 \end{aligned}$$

DISCUSSION OF MEASUREMENT ERRORS

There are three basic types of errors which can occur. Type I errors are related to limitations of the instrument design and tolerances of the instrument components. These are chiefly the meter linearity and resistor tolerances. Type II errors are due to basic probe accuracy tolerances, chiefly background signal, probe linearity, and variations in membrane temperature coefficient. Type III errors are related to the operator's ability to determine the conditions at the time of calibration. If calibration is performed against more accurately known conditions, Type III errors are appropriately reduced.

Individual Sources of Error

This description of sources of error can be used to attach a confidence to any particular reading of dissolved oxygen. The particular example given is for a near extreme set of conditions. As a generality, overall error is diminished when the probe and instrument are calibrated under conditions of temperature and dissolved oxygen which closely match the sample temperature and dissolved oxygen.

Type I

- A — is the error due to meter linearity
Error = +1% full scale of the measurement range.
- B — is the error due to tolerances in the instrument when transferring a reading from one range to another. Error = $\pm 1\%$ of the reading.

Type II

- A — errors due to probe background current
Error = $1.0\% \left(1 - \frac{\text{Meter Reading mg/l}}{\text{Calibration Value mg/l}} \right) \times \text{Calib. Value, mg/l}$
- B — errors due to probe non-linearity. Error = $\pm 0.3\%$ of reading.
- C — error caused by variability in the probe membrane temperature coefficient.
Error = zero if readings are taken at the calibration temperature
Error = $\pm 1\%$ of meter reading if readings are taken within 5°C of the calibration temperature.
Error = $\pm 3\%$ of meter reading for all other conditions.

Type III

- A — errors due to the accuracy of the instrument thermometer when used to measure the exact probe temperature during calibration.
Error = $\pm 1.5\%$ of reading.
- B — errors due to the assumption of mean barometric pressure.
Daily variation is usually less than 1.7% .
Error = $\pm 1.7\%$ of reading.
- C — errors assume an ability to estimate altitude to within ± 500 ft. when computing the altitude correction factor.
Error = $\pm 1.8\%$ of reading.

D — errors consider the possibility of only 50% relative humidity when calibrating the probe. If the actual relative humidity is 50% instead of 100%, the errors will be as follows:

Calibration Temperature \pm C	Error in percent of reading
0	(-) 0.3
10	(-) 0.6
20	(-) 1.15
30	(-) 2.11
40	(-) 3.60

Example of a Typical Error Calculation

The example given presumes the air calibration technique. If calibration is done with air saturated water, the relative humidity consideration (III-D) is eliminated. If the Winkler calibration method is used, Type III errors are deleted and replaced by the uncertainty attributable to the overall Winkler determination.

Data: Instrument calibrated at 25°C , elevation estimated at $2000' \pm 500'$, normal barometric pressure assumed, calibrated on $0-10$ mg/l range at 7.8 mg/l, readings taken on $0-20$ mg/l range at 10.5 mg/l at 8°C .

Type	Description	Calculations	Error mg/l
IA	Linearity	$= .01 \times 10.5 \text{ mg/l}$	$= .10$
IB	Range Change	$= .01 \times 10.5 \text{ mg/l}$	$= .10$
IIA	Probe Background	$= .01 \times \left(1 - \frac{10.5}{7.8} \right) 7.8 \text{ mg/l}$	$= .03$
IIIB	Probe Linearity	$= .003 \times 10.5 \text{ mg/l}$	$= .03$
IIIC	Temp. Compensation	$= .03 \times 10.5 \text{ mg/l}$	$= .31$
IIIA	Temp. Measurement	$= .015 \times 10.5 \text{ mg/l}$	$= .16$
IIIB	Pressure	$= .017 \times 10.5 \text{ mg/l}$	$= .18$
IIIC	Altitude	$= .18 \times 10.5 \text{ mg/l}$	$= .19$
IIID	R.H.	$= .016 \times 10.5 \text{ mg/l}$	$= .17$
	Maximum Possible Error		$= 1.27 \text{ mg/l}$
	Probable Error		$= \pm .63 \text{ mg/l}$

Considering a statistical treatment of the probable error at any time for any instrument, it is likely that the actual error in any measurement will be about $1/2$ of the possible error. In this case the probable error is about $\pm .5$ mg/l out of a reading of 10.5 mg/l, or 4.8% of the reading.

INSTRUMENT BATTERIES

Battery replacement or recharging on the YSI Model 54A is indicated if the "red line" adjustment cannot be made or O_2 calibration cannot be achieved. (Warning: a faulty probe will also not permit O_2 calibration.)

To replace batteries remove the four screws holding the rear cover of the instrument. The four batteries will be found on the battery terminal board inside. CAUTION: disconnect battery charger on YSI Model 54ARC before removing cover.

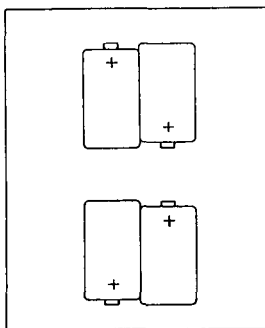


Figure 11

The YSI Model 54ARC contains four 1.25V Ni-Cd batteries (Burgess CD6 or equivalent). (See Figure 11). These batteries should be recharged when the instrument can no longer be red lined. Battery life should be three years or longer. Deeper discharge because of longer intervals between recharge will result in shorter battery life. The batteries should be recharged overnight, about 16 hours with the instrument off or 20 hours with the YSI Model 54ARC turned on.

The YSI Model 54ABP contains four 1.5V carbon-zinc (Panasonic UM-2N or equivalent). The life of these batteries is 1000 hours. Replace batteries every six months to minimize danger of corrosion due to dead or leaky batteries.

Battery holders are color coded. Positive (+ button) end of battery must go to red. (See Figure 11).

WARRANTY AND REPAIR

All YSI products carry a one-year warranty on workmanship and parts exclusive of batteries. Damage through accident, misuse, or tampering will be repaired at a nominal charge, if possible, when the item is returned to the factory or to an authorized YSI dealer.

If you are experiencing difficulty with any YSI product, it may be returned for repair, even if the warranty has expired. YSI maintains complete facilities for prompt servicing for all YSI products.

YELLOW SPRINGS INSTRUMENT CO., INC.
SERVICE DEPARTMENT
P.O. BOX 279
YELLOW SPRINGS, OHIO 45387, U.S.A.
PHONE: 513-767-7241

APPENDIX G-4

REDUCTION/OXIDATION (REDOX) POTENTIAL

FIELD MEASUREMENT OF OXIDATION-REDUCTION POTENTIAL

<u>Method:</u>	Electrometric
<u>Reference:</u>	Beckman Instruments, 1987
<u>Sensitivity:</u>	1 mV
<u>Optimum Range:</u>	-999.9 mV to +999.9 mV

Sample Handling: Determine on-site or within 4 hours

Reagents and Apparatus:

1. pH meter in absolute millivolt mode,
2. Platinum combination electrodes,
3. Beakers or plastic cups,
4. Certified pH buffer solutions, pH 4 and 7 saturated with a few crystals of quinhydrone,
5. Deionized water in squirt bottle.
6. All glassware soap and water washed, followed by two hot water rinses and two deionized water rinses.

Calibration:

1. Short the meter glass and reference inputs, and adjust the STANDARDIZE control until zero millivolts is displayed.
2. Place electrode in pH 4 buffer solution saturated with quinhydrone.

3. Record mV reading and compare to chart on Table 1.
4. Rinse electrode with deionized water and place in pH 7 buffer solution saturated with quinhydrone.
5. Record mV reading and compare to chart on Table 1.
6. If mV readings do not agree within ± 10 mV of the Table 1 values at the given temperature, follow electrode maintenance procedures described in the attached manual and recalibrate.

Procedure:

1. Calibrate meter using calibration procedure.
2. Pour the sample into a cleaner beaker or plastic cup.
3. Immerse electrode in solution allowing several minutes for meter to stabilize. Make sure the white AgCl junction on side of electrode is in the solution. The level of electrode solution must be approximately one inch above sample to be measured.
4. Rinse electrode with deionized water between samples. Recheck calibration with pH 4 buffer solution saturated with quinhydrone after every 5 samples.

Notes:

1. Eh is temperature and pH dependent. Therefore, the temperature and pH of samples should be measured at the same time as redox. For refrigerated or cool samples, use refrigerated buffers to calibrate meter.
2. Weak organic and inorganic salts and oil and grease are interferences in Eh measurements. If oil and grease are visible, note on data sheet. Clean electrode with soap and water, polish with scouring powder and rinse with distilled water. Then recalibrate meter.

3. Before going into the field:
 - a. Report any problems;
 - b. Do a quick calibration with quinhydrone saturated pH buffer solution to check electrode;
 - c. Prepare fresh quinhydrone saturated pH 4 and pH 7 solutions daily.

4. Following field measurements:
 - a. Report any problems;
 - b. Compare with previous data;
 - c. Clean all dirt off of meter and inside case;
 - d. Store electrode as follows;
 - 1) Slide rubber sleeve into position over the filling hole.
 - 2) Place cot over tip of electrode by threading platinum wire through opening and sliding cot onto glass body until porous plug is completely covered.

JDD/jkk/CAW
[wpmisc-600-39]
60776.05

Table 1

Redox Potential Calibration Chart

Quinhydrone Saturated pH 4 Solution

Temperature °C	20°	25°	30°
Theoretical Value (mV)	+268 mV	+263 mV	+258 mV

Quinhydrone Saturated pH 7 Solution

Temperature °C	20°	25°	30°
Theoretical Value (mV)	+92 mV	+86 mV	+79 mV

Instrument reading should be within ± 10 mV of Theoretical

[wpmisc-400-12]

BECKMAN

Φ™10 pH Meter

Φ™11 pH Meter

Φ™12 pH/ISE Meter

HSCE

WARARNTY

Your Φ^{TM} (pH^m) 10, 11, or 12 pH Meter is warranted to be free of manufacturing defects for one (1) year from the date of purchase. This does not include any damage to the equipment caused by misuse or abuse. Beckman Instruments, Inc., will, at Beckman's option, repair or replace your instrument with a new one. This is a limited warranty. You may have additional rights under your state laws. Statutes are not included in this warranty.

WARNING: This equipment generates, uses, and can radiate radio frequency energy and may cause interference to radio communications. Improper use, installation, or maintenance of the equipment may increase interference. It has been tested and found to comply with the limits for a Class A computing device pursuant to Subpart J of Part 15 of FCC Rules, which are designed to provide reasonable protection against such interference when operated in a commercial environment. However, the equipment in a residential area may cause interference, in which case the user should be advised that no assurance can be given that measures may be required to correct the interference.

Beckman Instructions 015-54400-9

BECKMAN

Φ^{TM} 10 pH Meter

Φ^{TM} 11 pH Meter

Φ^{TM} 12 pH/ISE Meter

pH MEASUREMENT

(Two standard method:
Condensed instructions)

I. SETUP

<p>①</p> <p>Prepare buffers (eg., pH 4 and 7).</p>	<p>②</p> <p>Prepare sample.</p>	<p>③</p> <p>Prepare de-ionized or distilled water for electrode rinse.</p>
<p>④</p> <p>(1) Omit reference if combination electrode is used. (2) ATC optional. Connect electrodes to instrument.</p>	<p>⑤</p> <p>Turn on and clear instrument.</p>	<p>⑥</p> <p>Display will read:</p>

II. STANDARDIZE

<p>①</p> <p>Rinse electrode(s). Blot excess.</p>	<p>②</p> <p>Immerse electrode(s) in STD 1. Stir briefly.</p>	<p>③</p> <p>Press PH, then STD.</p>	<p>④</p> <p>After STD stops flashing, display will read pH of STD 1.</p>
--	--	---	---

<p>⑤</p> <p>Rinse electrode(s). Blot excess.</p>	<p>⑥</p> <p>Immerse electrode(s) in STD 2. Stir briefly.</p>	<p>⑦</p> <p>Press STD.</p>	<p>⑧</p> <p>After STD stops flashing, display will read pH of STD 2.</p>
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III. MEASURE pH

<p>①</p> <p>Rinse electrode(s). Blot excess.</p>	<p>②</p> <p>Immerse electrode(s) in sample. Stir briefly.</p>	<p>③</p> <p>Press PH.</p>	<p>④</p> <p>After PH stops flashing, display will read pH of sample.</p>
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FOR MORE DETAILED INSTRUCTIONS ON pH MEASUREMENT, SEE NEXT PAGE.

MEASURING mV AND RELATIVE mV (Φ11, Φ12)

MEASURING CONCENTRATION (Φ12)

INSTRUMENT FUNCTIONS AND FEATURES

ELECTRODES, BUFFERS, AND ACCESSORIES

BATTERY REPLACEMENT, SERVICE
AND TROUBLESHOOTING

SPECIFICATIONS

pH MEASUREMENT: DETAILED INSTRUCTIONS

METHODS: The pH 10, 11, and 12 can measure pH from 0 to 15.99. They will perform one- or two-point standardization automatically, using any buffer listed below, at any temperature between -5°C and 100°C.

STANDARD pH BUFFERS RECOGNIZED BY THE pH 10, 11, AND 12:

1.68, 4.00, 7.00, 10.01, 12.45.

TWO-POINT STANDARDIZATION METHOD:

Two-point standardization, the preferred and more accurate method of pH measurement, should be used when pH accuracy of beyond ± 0.1 pH is required. Use buffers as close to the sample pH as possible, one above, and one below. (For example, if sample pH is about 8.5, use 7.00 and 10.01 pH buffers).

ONE-POINT STANDARDIZATION METHOD:

One-point standardization, a somewhat faster procedure, is recommended only if (a), accuracy of ± 0.1 pH unit is acceptable, and (b), sample pH is within 1.5 pH of that of the buffer used for standardization.

pH MEASUREMENT PROCEDURE:

1. Connect electrode(s) to appropriate input(s):
 - a. If a combination electrode is used, connect it to the input marked "pH".
 - b. If an electrode pair is used, connect the indicating electrode to the input marked "pH" and the reference electrode to the input marked "REF".
 - c. For better accuracy, or when measuring and/or standardizing at a temperature of other than 25°C, connect a Beckman 598115 Automatic Temperature Compensator probe to input marked "ATC".
2. Press **[0]** to turn on instrument, then press **[C]** to clear. Display will show [CH, A/UTC].
3. Rinse electrode(s) (and ATC if used) with deionized water. Blot excess.
4. Immerse electrode(s) (and ATC if used) in first standard. Stir briefly with electrodes to remove bubbles from electrode surfaces. Press **[M]**. Displayed pH value will have a resolution of 0.01. If 0.1 resolution is desired, press **[R]**.
5. Press **[M]**. When [cd] stops flashing, display will show [pH value locked, cd, > st, > st].
6. Rinse electrode(s) (and ATC probe if used) with deionized water. Blot excess. Proceed to appropriate step, according to desired type of standardization:
 - a. If ONE-POINT standardization is to be used, instrument is ready for sample measurement; proceed to Step 9.
 - b. If TWO-POINT standardization is desired, proceed to Step 7.
7. Immerse electrode(s) (and ATC if used) in second standard. Stir briefly with electrodes to remove bubbles from electrode surfaces. Press **[M]**. When [cd] stops flashing, display will show [pH value locked, cd, > st, > st].
8. Rinse electrode(s), (and ATC probe if used) with deionized water. Blot excess.
9. Immerse electrode(s) (and ATC if used) in sample. Stir briefly with electrodes. Press **[M]**. When [cd] stops flashing, display will show [pH value locked, cd]. Measurement is now complete. Repeat Steps 8 and 9, above, for additional samples.
10. If continuous pH monitoring is desired, press **[M]** to turn off Auto Read function.

MEASURING mV AND RELATIVE mV (Φ11, Φ12)

MEASURING CONCENTRATION (Φ12)

INSTRUMENT FUNCTIONS AND FEATURES

ELECTRODES, BUFFERS, AND ACCESSORIES

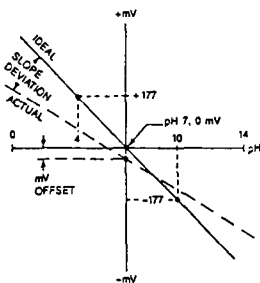
BATTERY REPLACEMENT, SERVICE AND TROUBLESHOOTING

SPECIFICATIONS

pH MEASUREMENT: PRINCIPLES AND THEORY

The pH 10/11/12 pH Meter is essentially a high-impedance voltmeter with a microcomputer that translates voltage and temperature data into pH units. At 25°C, the ideal pH electrode system develops -59 mV per pH unit increase, with 7.00 pH = 0 mV.

Standardization allows the meter to compensate for non-ideal electrode characteristics. One-point standardization compensates for millivolt offset; two-point standardization compensates for both millivolt offset and slope deviation. See diagram below.



Ideal and Actual Electrode Response Compared

The pH 10, 11, and 12 may be standardized with any of five standard pH buffers: 1.68, 4.00, 7.00, 10.01, and 12.45. Standardization may be accomplished with any two buffers, used in any order (and at any temperature, if ATC is used). When STANDARD is pressed, the instrument automatically recognizes the buffer. The relationship between pH and electrode voltage changes with temperature. For precise pH measurements or temperatures not close to 25°C, a Beckman 588115 ATC (Automatic Temperature Compensator) probe should be used. With this probe, the instrument automatically compensates for the temperature characteristics of the buffer, permitting a sample to be measured at any temperature, even if different from the buffer temperatures. With ATC, the instrument measures and displays temperatures from -5°C to 100°C. If an ATC probe is not used, the instrument defaults and displays 25°C.

The pH calculation is based on the Nernst equation:

$$E = E_0 - \frac{2.3 RT}{nF} \log a_i$$

E is the total potential, in millivolts, developed between the sensing and reference electrodes; E_0 varies with the choice of electrodes, temperature, and pressure; $2.3RT/nF$ is the Nernst factor (R and F are constants, n is the charge on the ion, including sign, T is the temperature in degrees Kelvin), and a_i is the activity of the ion to which the electrode is responding.

For further information on principles and theory of pH measurement, refer to The Beckman Handbook of Applied Electrochemistry (Beckman Bulletin 7729).

MEASURING mV AND RELATIVE mV (Φ11, Φ12)

MEASURING CONCENTRATION (Φ12)

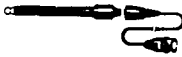

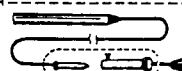


INSTRUMENT FUNCTIONS AND FEATURES


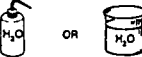


ELECTRODES, BUFFERS, AND ACCESSORIES

BATTERY REPLACEMENT, SERVICE AND TROUBLESHOOTING

SPECIFICATIONS

FOR ACCURATE mV MEASUREMENTS WITH THE $\Phi 11$ OR $\Phi 12$, THE FOLLOWING ITEMS ARE RECOMMENDED:

1.		pH indicating electrode, Futura II. Futura II cable with BNC connector. NOTE: If combination pH electrode is used, omit separate reference electrode (Item 2, below).
OR		
1a.		Metallic electrode with 2 mm pin connector + pin-to-BNC adaptor.
OR		
1b.		Ion-Selective electrode with BNC connector (or U.S. standard connector + U.S. standard-to-BNC adaptor).
2.		Reference electrode, Futura II. Cable with 2 mm pin connector, Futura II. NOTE: Omit reference electrode if combination pH electrode is used.
3.		Standard solution(s) appropriate to the application.

4.		Clean beaker(s) or equivalent container(s), 100-250 mL, for containing standard solution(s).
5.		Squirt bottle or beaker containing deionized or distilled water for rinsing electrodes.
6.		Clean towels, "Kimwipes™", etc., for blotting electrodes.
7.		The sample to be measured.

For part numbers, see "Electrodes, Buffers, and Accessories."
For mV measurement procedures, see next page.

MEASURING mV AND RELATIVE mV ($\Phi 11$, $\Phi 12$)

MEASURING CONCENTRATION ($\Phi 12$)



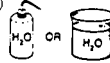



INSTRUMENT FUNCTIONS AND FEATURES

ELECTRODES, BUFFERS, AND ACCESSORIES





BATTERY REPLACEMENT, SERVICE
AND TROUBLESHOOTING

SPECIFICATIONS









I. SETUP

<p>①</p>  <p>STANDARD</p> <p>Prepare standard solution.</p>	<p>②</p>  <p>SAMPLE</p> <p>Prepare sample.</p>	<p>③</p>  <p>H₂O OR H₂O</p> <p>Prepare deionized or distilled water for electrode rinse.</p>
<p>④</p>  <p>REF pH ATC</p> <p>Connect electrodes to instrument.</p>	<p>⑤</p>  <p>Turn on and clear instrument.</p>	<p>⑥</p> <p>Display will read:</p> 

II. mV MEASUREMENT, ABSOLUTE. FOR RELATIVE mV MEASUREMENT, SEE III, BELOW.

<p>①</p>  <p>Rinse electrode(s). Blot excess H₂O.</p>	<p>②</p>  <p>STANDARD OR SAMPLE</p> <p>Immerse electrode(s) in standard or sample. Stir briefly.</p>	<p>③</p>  <p>mV</p> <p>Press mV.</p>	<p>④</p> <p>After mV stops flashing, display will read absolute mV of solution.</p> 
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For RELATIVE mV Measurement,
Proceed with following steps:

III. mV MEASUREMENT, RELATIVE			
<p>①</p>  <p>Rinse Electrodes. Blot excess.</p>	<p>②</p>  <p>STANDARD</p> <p>Immerse electrodes in standard solution to be used to establish zero mV point. Stir briefly.</p>	<p>③</p>  <p>mV STD</p> <p>Press mV then STD.</p>	<p>④</p> <p>After mV stops flashing, display will read 000.0 mV REL mV</p> 
<p>⑤</p>  <p>Rinse electrodes. Blot excess.</p>	<p>⑥</p>  <p>SAMPLE</p> <p>Immerse electrodes in sample. Stir briefly.</p>	<p>⑦</p>  <p>mV</p> <p>Press mV.</p>	<p>⑧</p> <p>After mV stops flashing, display will read mV relative to the standard.</p> 

NOTE
INSTRUMENT WILL REMAIN IN RELATIVE mV MODE UNTIL
EITHER **mV**, **STD**, OR **1** IS PRESSED.

FOR MORE DETAILED INSTRUCTIONS ON mV MEASUREMENT, PROCEED TO NEXT PAGE.

MEASURING CONCENTRATION (§12)

INSTRUMENT FUNCTIONS AND FEATURES

ELECTRODES, BUFFERS, AND ACCESSORIES

BATTERY REPLACEMENT, SERVICE
AND TROUBLESHOOTING

OPERATIONS

mV MEASUREMENT: TYPICAL USES

Some uses of the mV mode are monitoring chemical reactions, quantifying ions, and determining the oxidizing-reducing potential (ORP) of a given sample. Because such measurements are usually not specific for a particular ion or species, readings must be interpreted carefully to obtain meaningful results. The user should have an understanding of the reaction that is occurring, or is desired, and of any sample components that could potentially interfere. For more detailed information, refer to the Beckman Handbook of Applied Electrochemistry (Beckman Bulletin 7739).

The mV mode may also be used with ion-selective electrodes. The relative mV mode can be used in the standard addition or standard subtraction method of ion analysis.

STANDARD SOLUTION(S)

Make up appropriate standard solution(s) to provide known voltage(s), depending on the reference electrode used and the temperature. For example, common standards used in redox measurements are pH 4 and pH 7 buffers saturated with quinhydrone.

mV MEASUREMENT PROCEDURE

1. Connect electrodes to appropriate inputs:
 - a. Connect indicating electrode to input marked "PH". A Pin-to-BNC Adaptor may be required as most metallic electrodes have a pin connector.
 - b. Connect reference electrode to input marked "REF".
2. Press **[0]** to turn on instrument, then press **[C]** to clear. Display will show [Clr. AUTO].
3. Rinse electrodes with deionized water. Blot excess.
4. Immerse electrodes in desired solution. Press **[mV]**. Displayed value is absolute mV, as indicated by display of **[mV]**. When **[mV]** stops flashing, display will show [mV reading locked, **[mV]**].

RELATIVE mV MEASUREMENT PROCEDURE

1. Perform Steps 1 through 3 of mV MEASUREMENT PROCEDURE, above.
2. Immerse electrodes in standard solution to be used to establish the zero mV point. Press **[mV]**. When **[mV]** stops flashing, display will read [000.0 mV]. Note that, in mV mode, pressing **[mV]** causes the instrument to establish the zero mV point at the value of the current reading. If desired, this step may be repeated at any time to re-establish the zero mV point.
3. Rinse electrodes with deionized water. Blot excess.
4. Immerse electrodes in sample. Press **[mV]**. Displayed value is relative mV, as indicated by display of [REL mV]. When **[mV]** stops flashing, display will show [sample relative mV value locked, **[mV]**]. Absolute mV reading of the standard solution is automatically subtracted from the absolute mV reading of the sample, resulting in a relative mV reading for the sample.
5. If continuous readout of relative mV is desired, press **[mV]** to turn off Auto Read function.

NOTE

VOLTAGE DIFFERENCE BETWEEN STANDARD SOLUTION AND SAMPLE MUST NOT EXCEED 1000 mV. MAXIMUM DISPLAY RANGE IN mV MODE IS ± 999.9 mV.

NOTE

IN mV MODE, THE 558115 AUTOMATIC TEMPERATURE COMPENSATOR PROBE MAY BE USED FOR TEMPERATURE MEASUREMENT AND DISPLAY, BUT DOES NOT HAVE ANY TEMPERATURE-COMPENSATING EFFECT.

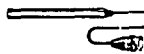
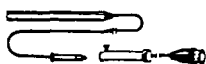
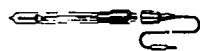
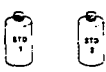
MEASURING CONCENTRATION (§12)


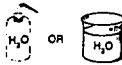


INSTRUMENT FUNCTIONS AND FEATURES

ELECTRODES, BUFFERS, AND ACCESSORIES

BATTERY REPLACEMENT, SERVICE
AND TROUBLESHOOTING

FOR ACCURATE CONCENTRATION MEASUREMENTS WITH THE $\Phi 12$,
THE FOLLOWING ITEMS ARE RECOMMENDED:

1.  Ion-Selective electrode with BNC connector.
OR
 - 1a.  Ion-Selective electrode with U.S. standard connector + U.S. standard-to-BNC adaptor.
 2.  Reference electrode, Futura II.
Futura II cable with 2 mm pin connector.
NOTE: Depending on the application, a salt-bridge or double-junction electrode may be required.
 3.  Two standard solutions of appropriate concentration, selected from the following values: 100, 250, 500, 1000, 2500, 5000, 10000, 25000, 50000, and 100000 units. Concentration can be expressed in any desired units such as ppm, mM, mg/L, and $\mu\text{g/L}$.
Make up these solutions per procedure or by diluting a stock solution to suit your requirement.
- NOTE
CONCENTRATION UNITS FOR BOTH STANDARD SOLUTIONS MUST BE THE SAME AS DESIRED FOR SAMPLE READINGS.

4.  Two clean beakers or equivalent containers, approximately 100-250 mL, for containing the two standard solutions.
5.  Squirt bottle or beaker containing deionized or distilled water for rinsing electrodes.
6.  Clean towels, "Kimwipes"TM, etc., for blotting electrodes.
7.  The sample to be measured.

For part numbers, see "Electrodes, Buffers, and Accessories."
For concentration measurement procedure, see next page.

MEASURING CONCENTRATION ($\Phi 12$)

INSTRUMENT FUNCTIONS AND FEATURES

ELECTRODES, BUFFERS, AND ACCESSORIES

BATTERY REPLACEMENT, SERVICE AND TROUBLESHOOTING

I. SETUP

<p>①</p> <p>Prepare standard solutions (e.g., 5.00 and 10.00 units).</p>	<p>②</p> <p>Prepare sample.</p>	<p>③</p> <p>Prepare deionized or distilled water for electrode rinse.</p>
<p>④</p> <p>Connect electrodes to instrument.</p>	<p>⑤</p> <p>Turn on and clear instrument.</p>	<p>⑥</p> <p>Display will read:</p>

II. STANDARDIZE

<p>①</p> <p>Rinse electrodes. Blot excess H₂O.</p>	<p>②</p> <p>Immerse electrodes in Standard 1. Stir briefly.</p>	<p>③</p> <p>Press CONC, then press STD 1 repeatedly until display shows Standard 1 value, e.g., 5.00.</p>	<p>④</p> <p>When STD starts flashing, proceed.</p>
<p>⑤</p> <p>Rinse electrodes. Blot excess.</p>	<p>⑥</p> <p>Immerse electrodes in Standard 2. Stir briefly.</p>	<p>⑦</p> <p>Press STD 2 repeatedly until display shows Standard 2 Value, e.g., 10.00.</p>	<p>⑧</p> <p>When STD starts flashing, proceed.</p>

III. MEASURE CONCENTRATION

<p>①</p> <p>Rinse electrodes. Blot excess.</p>	<p>②</p> <p>Immerse electrodes in sample. Stir briefly.</p>	<p>③</p> <p>Press CONC.</p>	<p>④</p> <p>After STD starts flashing, display will read concentration of sample.</p>
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FOR MORE DETAILED INSTRUCTIONS ON CONCENTRATION MEASUREMENT, PROCEED TO NEXT PAGE.

INSTRUMENT FUNCTIONS AND FEATURES
ELECTRODES, BUFFERS, AND ACCESSORIES

BATTERY REPLACEMENT SERVICE

The following procedure, utilizing two-point standardization, can be used to measure concentrations of ions in almost any desired units.

STANDARD SOLUTIONS:

Standards can be made from any type of solution, with concentrations selected from the following values: 1.00, 2.50, 5.00, 10.00, 25.0, 50.0, 100.0, 250.0, 500, and 1000 units.

Units of concentration may be any that the user finds convenient. CONCENTRATION UNITS FOR BOTH STANDARD SOLUTIONS MUST BE THE SAME AS DESIRED FOR SAMPLE READINGS.

Some examples of units are: parts per million, percent, moles per liter, parts per billion, milliequivalents per liter, and ounces per gallon.

Select two standard values as close as possible to the anticipated sample value, preferably with one standard value below and one standard value above the sample. For example, if sample solution is about 150 millimoles per liter (mM), make up standards of 100 mM and 250 mM. If sample concentration varies widely, for example, between 10 mM and 75 mM, make up standards of 10 mM and 100 mM.

Standards and samples should be at the same temperature to avoid temperature-dependent variations in readings.

NOTE

Standard and sample solutions may require ionic strength adjustment or interfering ion removal. Consult electrode instructions for details.

CONCENTRATION MEASUREMENT PROCEDURE:

1. Connect electrodes to appropriate inputs:
 - a. Connect ion-selective electrode to input marked "ISE".
 - b. Connect reference electrode to input marked "REF".

NOTE

If, in addition to the ion-selective electrode, a combination pH electrode is connected to the instrument AND is to be immersed in the same solution, DO NOT use a separate reference electrode.

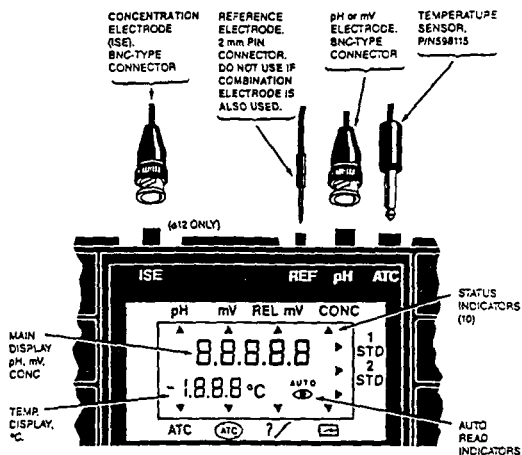
2. Press **[0]** to turn on instrument, then press **[1]** to clear. Display will show [Cl; AUTO].
3. Rinse electrodes with deionized water. Blot excess.
4. Immerse electrodes in first standard solution. Press **[MODE]**, then press **[7]** as many times as needed for the concentration value of the first standard to show on the display. When **[7]** is pressed repeatedly, the display steps through the following values: 1.00, 2.50, 5.00, 10, 25, 50, 100, 250, 500, and 1000 concentration units. For example, if the concentration of the standard is 100 units, press **[7]** seven times and the display will show [100]. When [**CD**] stops flashing, display will show [100 locked. **CD** - > **SH**].
5. Rinse electrodes with deionized water. Blot excess.
6. Immerse electrodes in second standard solution. The first and second standards must be different, but can be measured on any scale. Press **[7]** as many times as needed for the display to show the concentration value of the second standard, e.g., 250. When [**CD**] stops flashing, display will show [250 locked. **CD** - > **SH**].
7. Rinse electrodes with deionized water. Blot excess.
8. Immerse electrodes in sample. Press **[MODE]**. When [**CD**] stops flashing, display will show [sample value locked. **CD**]. Measurement is now complete. Repeat Steps 7 and 8, above, for additional samples.
9. If continuous concentration readout is desired, press **[MENU]** to turn off Auto Read function.

INSTRUMENT FUNCTIONS AND FEATURES

ELECTRODES, BUFFERS, AND ACCESSORIES

BATTERY REPLACEMENT, SERVICE

ELECTRODE CONNECTIONS



DISPLAY FEATURES AND STATUS INDICATORS

DISPLAY

Main Display

1. Readout of the measured variable: pH, mV, or concentration.
2. [C] is displayed, indicating that instrument is cleared, when [C] is pressed.
3. Error message:

[E] indicates an excessive, potentially damaging, input voltage, typically caused by static electricity when the electrode pair is not in solution. In this case, immerse electrodes in solution, press [C], and proceed with measurement. If [E] again appears, check connections and electrodes for possible open circuit.

Temperature Display (°C)

The small digits display temperature in °C. Will read 25°C if ATC not plugged in.

AUTO

AUTO READ ON/OFF indicator for AUTO READ ON/OFF Key, described subsequently.

⊙

AUTO READ Status Indicator (eye symbol). Functions during standardization and when instrument is in AUTO mode. During standardization, the eye symbol starts flashing when [C] is pressed, and locks on when the reading has stabilized. During sample measurement in AUTO mode, the eye symbol starts flashing when a mode key is pressed, and locks on when the reading has stabilized. The reading remains locked until a mode key is pressed. If an interval of approximately 30 minutes elapses without a key being pressed, the instrument turns off automatically to conserve the batteries, but retains all standardization data in memory.

STATUS INDICATORS

ATC Indicates that ATC is plugged in. The instrument measures and displays temperature within the range of -5°C and 100°C. Display of [E] indicates that the temperature sensed is outside the measurement range, or the ATC is nonfunctional.

⊙

Indicates that ATC is not plugged in. The temperature reading defaults to 25°C.

pH

▲

Indicates that instrument is in pH mode.

mV

▲

Indicates that instrument is in mV mode.

REL

▲

Indicates that instrument is in relative mV mode.

CONC

▲

Indicates that instrument is in concentration mode. #12 only.

1

STD

Indicates that one standard has been used to standardize for the selected measurement mode (pH or CONC).

2

STD

Indicates that two standards have been used to standardize for the selected measurement mode (pH or CONC).

?

///

Indicates a questionable electrode and/or standardization.

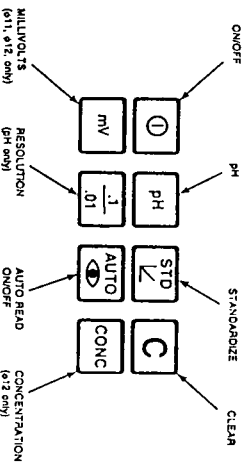
⊙

Indicates that batteries should be replaced.

INSTRUMENT FUNCTIONS AND FEATURES

ELECTRODES, BUFFERS, AND ACCESSORIES

KEYPAD



KEYPAD FUNCTIONS

KEY

ON/OFF

Instrument ON/OFF Key. When OFF, the instrument retains the standardization data in memory. Instrument should be automatically after 30 minutes of inactivity. AUTO READ is ON. (See below.)

STD

Clear Key. Clears instrument, resetting all standardization data to default values, and returning instrument to AUTO mode.

AUTO

- When Auto Read is ON:
 - The instrument turns the display ON and returns on the display.
 - The instrument uses the selected signal for stability. During this time, the display shows the selected signal. The instrument will not perform a standardization until the signal has met the stability requirement (see SPECIFICATIONS). (ON) remains on continuously, and the digital display locks onto the reading. No further measurements are made until a key is pressed.
 - When the instrument is in the standardization mode, the instrument turns off automatically and retains all standardization data.
- When Auto Read is OFF:
 - [AUTO] disappears from display.
 - The instrument continuously measures and displays in the selected mode: pH, mV, or CONCENTRATION.
 - After 1 to 2 hours without keypad input, the instrument turns off automatically but retains all standardization data.

1/0.1

pH Resolution Selection Key. Changes resolution of the display: pH reading from 0.01 to 0.1 pH units, or 0.1 to 1.0 pH units. For resolution (0.1), time required to do Auto Read is less, is shorter. (See SPECIFICATIONS).

mV

Selects the pH measurement mode.

PH

Selects the mV mode (pH 11, pH 12). Key measurement of either activity or relative mV. See MEASURING mV AND RELATIVE mV.

CONC

Selects the concentration measurement mode (pH 12). Used with specific ion electrodes.

STD

Standardize Key. Standardizes instrument. Depends upon mode.

- pH Mode: [STD] key causes the instrument to automatically identify the pH value of the buffer from any one of the following: 1.68, 4.00, 7.00, 10.01, and 12.46.
- Concentration Mode: [STD] key causes the instrument to establish the appropriate level at the pH 11 or 12.
- CONC Mode (pH 12): Repeated pressing of [STD] key causes the instrument to stop through the following sequence of values: 120, 250, 500, 10, 25, 50, 250, 500, and 1000 concentration units.

COMBINATION ELECTRODES:

	Standard 5" x 1/2"	Probe 8-10" x 3/8"	Test-Tube 8-9" x 5-6mm
Glass Body Ag/AgCl, Refillable	39520	39521	39522
Glass Body Calomel, Refillable	39527	39528	39529, 39528 (7")
Epoxy Body Calomel, Refillable	39836	—	39839
Epoxy Body Ag/AgCl, Refillable	39831	39833	39835
Epoxy Body Ag/AgCl, Gel Filled	39838	—	39834
Epoxy Body, Star Ag/AgCl Refillable	39837	—	—
Glass Body, Star Ag/AgCl Refillable	39524	—	—
Flat Bulb, Epoxy Body	39523	—	—

ELECTRODE PAIRS:

pH INDICATING ELECTRODES:

0-14 pH, Spherical Bulb	39314
0-11 pH, Dome Bulb (Durable)	39316

METALLIC ELECTRODES:

Silver/Billet	39251
Platinum Inlay	39273

REFERENCE ELECTRODES:

Calomel Half Cell, Quartz Fiber Junction	39416
Calomel Half Cell, Ceramic Pin Junction	39417
Ag/AgCl Half Cell, Quartz Fiber Junction	39418
Calomel Half Cell, Sleeve Double Junction	39419
Calomel Half Cell, Inverted Sleeve Junction	39420
Ag/AgCl Half Cell, Inverted Sleeve Junction	39421

2. FUTURA II KEEPER CABLES

COMBINATION AND INDICATING
ELECTRODE CABLES

1m, BNC Connector	597578
2m, BNC Connector	597579
6m, BNC Connector	597580

REFERENCE ELECTRODE
KEEPER CABLES

1m, 2mm Pin Connector	598982
2m, 2mm Pin Connector	598983
6m, 2mm Pin Connector	598984

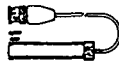
3. SALT BRIDGE: 563853

4. SUBMERSIBLE COMBINATION pH ELECTRODE WITH ATC: 39530

5. AUTOMATIC TEMPERATURE COMPENSATOR, 598115:

Permits temperature measurement and display, and temperature compensation of pH and ion-selective electrodes, within range of -5°C to 100°C. Epoxy body. For use with standard 5-inch (13-cm) electrodes. Includes 39" (1 meter) cable with miniature phone jack.

6. ELECTRODE ADAPTORS:



592362 Standard to
BNC Adaptor



592367 PIN to BNC Adaptor

Adapts Glass Electrode (GE) BNC terminal on
pH Series pH Meters to accommodate
electrodes with U.S. Standard Connectors.

Adapts Glass Electrode (GE) BNC terminal on
pH Series pH Meters to accommodate
electrodes with Pin Connectors.
Typically used to connect metallic electrodes.

7. BUFFERS

	4 Pack of Pints	1 Gallon	5 Gallons	Powder (Colorless)
pH 4 Buffer (red)	582517	566001	582522	3005
pH 7 Buffer (green)	582521	566003	582523	3007
pH 10 Buffer (blue)	582525	566005	582524	3019
pH 12.45				3010

8. FILLING SOLUTIONS

Description	Quantity	Part No.
Combination Electrode Filling Solution or Ag/AgCl Reference Electrode Filling Solution (4M KCl/AgCl saturated; to be used with Ag/AgCl internals)	4-pack of 100 mL bottles	566467
Reference Electrode Filling Solution (saturated KCl to be used with Calomel internals)	4-pack of 100 mL bottles	566468
Electrode Soaking Solution	4-pack of 100 mL bottles	566578
Salt Bridge Solution, Contains Sodium Nitrate and Sodium Acetate	4-pack of 100 mL bottles	566469
Filling Solution, 1M, KCl Saturated with AgCl (Star-Series electrodes only)	4-pack of 100 mL	568943

9. pH START-UP KIT:

39831 Electrode, Cable, Thermocompensator, Sample Buffers, Filling solution	Part No. 123105
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10. pH STAND LAB ORGANIZER

11. pH DELUXE FIELD CASE

12. pH SOFT CASE

13. pH MOUNT, WALL/SHELF BRACKET


Your #10, 11, or 12 is powered by two 3.6 volt lithium batteries. Expected battery life is over 1,000 hours of continuous operation. Replacement batteries can be obtained by ordering Part No. 945574 from your local Beckman office. (In U.S. call 1-800-742-2343).

Acceptable replacement batteries are also available on a world-wide basis:

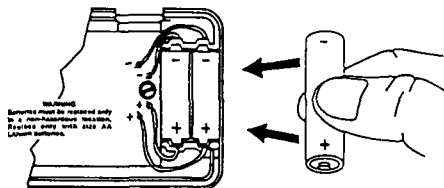
Mfr.	Part No.
Electrochem Industries	36940TC
Power Conversion Inc.	TC8-41
Salt Advanced Battery Div.	LS8
Tadiran	TL-2100 AAS

Local suppliers may be found in your telephone directory.


Note that these batteries are 3.6 volt lithium cells. Do not attempt to replace them with 1.5 volt alkaline or carbon-zinc cells.

If instrument display indicates low battery voltage  or if display is blank when instrument is turned on, batteries should be replaced:



1. Remove 2 Phillips screws and bottom cover from instrument.
2. Lift out old batteries.
3. Note (+) and (-) markings in battery compartment.
4. Check (+) and (-) markings on batteries and insert as shown:





5. Replace back cover and screws.

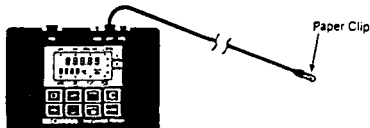
IMPORTANT: A "BREAK-IN" PERIOD OF UP TO 30 MINUTES IS REQUIRED WHEN SOME NEW LITHIUM BATTERIES ARE FIRST PLACED INTO SERVICE. DURING THIS PERIOD, THE LOW BATTERY SYMBOL AND SOME "GHOSTING" MAY APPEAR ON THE DISPLAY. IF SO, LEAVE INSTRUMENT ON FOR 20-30 MINUTES AND THEN PRESS . THE LOW BATTERY SYMBOL AND "GHOSTING" SHOULD DISAPPEAR.




TROUBLESHOOTING PROCEDURE

1. Disconnect electrode cable(s) from instrument. Press  to turn on instrument, then press  to clear. Display should show [Ck: AUTO]. If not, replace batteries per BATTERY REPLACEMENT, above. If instrument is still inoperative, call Service Hot Line: 1-800-662-6217.
2. Insert one end of a paper clip into the small hole in the center of the "pH" input connector. Hold the other end of the clip to the inside barrel of the same connector as shown.



3. Press  then . The display should lock at pH 7.00, indicating a one-point standardization.
 - a. If instrument passes test, go to Step 4.
 - b. If instrument fails test, call Service Hot Line: 1-800-662-6217.
4. Reconnect pH electrode cable to "pH" input connector. Short the input connector of the cable.



Press  then . Display should lock at pH 7.00. Press  then remove paper clip. Reading should drift.

- a. If instrument passes test, go to Step 5.
- b. If instrument fails test, call Beckman Electrochemistry Applications:
 - 1-800-854-8067 Outside California
 - 714-871-4848 Within California
5. Reconnect pH electrode(s). Immerse electrode(s) in pH 4 buffer and perform one-point standardization. Then immerse electrode(s) in pH 10 buffer and take pH reading. At 25°C, the reading should be between 8.7 and 10.1 pH.
 - a. If the test is passed, the pH meter, cable, and electrode(s) are functioning properly.
 - b. If test is failed, the pH electrode(s) must be rejuvenated or replaced. The electrode rejuvenation

INSTRUMENT BECKMAN PART NO.	pH 10 123132	pH 11 123133	pH 12 123134
pH MEASUREMENT			
Range	0 to 15.99 pH	0 to 15.99 pH	0 to 15.99 pH
Resolution (Selectable)	0.01, 0.1 pH unit	0.01, 0.1 pH unit	0.01, 0.1 pH unit
Relative Accuracy	± 0.01 pH	± 0.01 pH	± 0.01 pH
Auto Read Mode	0.1 pH Resolution: Display locks after reading is stable within 1.0 mV for 4 seconds. 0.01 pH Resolution: Display locks after reading is stable within 0.5 mV for 8 seconds.		
Buffers Recognized by Instrument: 1.68, 4.00, 7.00, 10.01, 12.45 pH.			
MILLIVOLT MEASUREMENT			
Range	—	-999.9 to +999.9 mV	-999.9 to +999.9 mV
Resolution	—	0.1 mV	0.1 mV
Accuracy	—	± 0.2 mV ± 0.02% of reading, relative mV	± 0.2 mV ± 0.02% of reading, relative mV
Auto Read Mode	—	Display locks after reading is stable within 0.5 mV for 8 seconds.	
CONCENTRATION MEASUREMENT			
mV Accuracy	—	—	± 0.1 mV
Auto Read Mode	—	—	Display locks after reading is stable within 0.25 mV for 8 seconds.
Useable Standard Values	—	—	Two values, in any desired units, selected from the following: 1.0, 2.5, 5.0, 10, 25, 50, 100, 250, 500, and 1000.

TEMPERATURE MEASUREMENT (ALL MODELS)

Range: -5°C to 100°C
Resolution: 0.1°C
Accuracy (with Beckman 598115 Probe): ± 0.5°C

MISCELLANEOUS (ALL MODELS)

Input Connections:
1. BNC input for pH, mV, and concentration.
2. 2-mm pin connector for reference electrode.
3. Miniature phone jack for Beckman 598115 Automatic Temperature Compensator.

Operating Temperature: 15°C to 40°C, ambient, non-condensing.
Power Source: Two lithium cells, 3.6 volts each, AA Size.

Error Indications:
1. Input overvoltage (all modes)
2. Temperature compensation non-functional
3. Low batteries
4. Questionable electrode/standardization.

Size: 5.2 inches x 3.8 inches x 1.3 inches.

*Beckman Co
National
7 West Poplar Ave
- 1st, CT 93257*

Instructions

E-11

Platinum Combination Electrode

Catalog No. 13-639-82

The Fisher platinum combination electrode combines a silver/silver chloride reference element and a platinum-wire indicating element in a single probe. This "dual element" configuration eliminates the need for two separate electrodes and is especially applicable to measurements in narrow-neck flasks and other restricted-entry receptacles.

The platinum combination electrode is recommended for use with automatic titrators and similar electroanalytical equipment. The close physical proximity of the porous-plug liquid junction to the platinum-wire indicator section results in reduced resistance between these elements and produces a rapid dynamic response for both redox measurements and potentiometric titrations. Additionally, the electrode is ideally suited for any application that involves the measurement of oxidation-reduction potentials or requires the use of a "noble metal" sensor.

The electrode measures 5 inches with a 30-inch lead, and functions over a -5° to 110°C temperature range. The filling solution is 4M KCl saturated with AgCl (Fisher No. So-P-135), and flow rate at the junction is less than $8\ \mu\text{l}$ per hour at an 8 cm head. Reference output is 44 \pm 1mV vs. S.C.E., while junction resistance is less than 10K ohms.

INSTALLATION

Place the platinum combination electrode into service as follows:

1. Remove cap from supplied filling-solution bottle, and screw on dispenser spout.
2. Lower rubber sleeve on electrode body until filling hole is exposed, and fill reference cavity with electrolyte until meniscus reaches a level approximately $\frac{1}{8}$ -inch below filling hole.

NOTE: Always use 4M KCl solution saturated with AgCl (Fisher No. So-P-135) as the electrolyte. NEVER USE SATURATED KCl FILLING SOLUTION.

3. Place electrode upright in empty beaker to permit filling solution to wet and flow through porous plug, as evidenced by formation of KCl crystals on outer surface of plug.

NOTE: If no flow is observed within 30 minutes, or if response is unsatisfactory during an analysis, soak electrode in dilute KCl (0.1M) for several hours, and then perform the following procedure:

- a. Hold electrode (cap up) at a 45° angle between thumb and forefinger on left hand, so that filling hole faces out and is directly opposite base of thumb.
- b. Insert dispensing spout into filling hole.
- c. Make sure that electrode is supported by base of thumb, then firmly press spout into filling hole to make an airtight seal.

NOTE: Normally, spout tip will not touch internal element; while applying pressure, however, care should be exercised to prevent contact. If necessary, cut off a portion of the tip.

- d. While maintaining seal, squeeze filling bottle firmly so

that electrode becomes pressurized.

NOTE: A bead of liquid should form at liquid junction in about 30 seconds; in some cases, however, it may be necessary to maintain pressure for several minutes. If flow cannot be established, refer to REJUVENATION section.

4. Mount electrode on suitable holder and connect jacks to pH meter.

OPERATION

For optimum operation with the platinum combination electrode, observe the following general procedures:

1. Rubber sleeve should always be lowered on electrode body to expose filling hole and permit proper electrolyte leakage.
2. Level of electrolyte must always be maintained above surface of sample solution to avoid backflow of sample into electrolyte. Refill reference cavity as required.
3. After removing electrode from one solution and before immersing in another, the outer surface should be rinsed with distilled water.

STORAGE

When not in use, store the platinum combination electrode as follows:

1. Slide rubber sleeve into position over the filling hole.
2. Place supplied cap over tip of electrode by threading platinum wire through opening and sliding cap onto glass body until porous plug is completely covered.

REJUVENATION

Rejuvenation of the platinum combination electrode may only require a simple cleaning. Occasionally, a more thorough cleaning is required, or the porous-plug junction may have to be unblocked. Each is covered separately below.

Simple Cleaning

A simple cleaning of the electrode is done as follows:

1. Wash electrode surface with a good detergent.
NOTE: RBS-25 detergent (Fisher No. So-C-181) is recommended.
2. Polish platinum wire with scouring powder.
3. Rinse electrode thoroughly with distilled water.

Thorough Cleaning

For a more thorough cleaning, perform the following:

1. Connect tip of large cable plug to negative terminal of a 22V dry cell, then immerse tip of electrode in a 1N solution of hydrochloric acid.
2. Similarly connect a platinum or graphite electrode to positive terminal of dry cell and immerse tip of electrode in same solution.

NOTE: Hydrogen will evolve rapidly, and the metallic electrode will be cleaned by electrolysis in 5 to 10 seconds.

3. After cleaning, disconnect both electrodes and rinse



each with distilled water

Unblocking the Junction

If the liquid junction should become partially blocked, perform the following:

1. Inspect reference cavity for crystallization.
2. If crystals are evident, proceed as follows:
 - a. Remove filling solution by shaking it out through filling hole.
 - b. Rinse cavity repeatedly with distilled water until all crystals are dissolved.
 - c. Refill cavity with fresh 4M KCl solution saturated with AgCl (Fisher No. So-P-135).

CAUTION: Never use saturated KCl as the electrolyte.

- d. Repeat all of step 3 under INSTALLATION.
3. If difficulty persists, perform the following in sequence depending upon the severity of the blockage:
 - a. Soak electrode overnight in dilute KCl (0.1M).
 - b. Boil junction in dilute KCl for 5 to 10 minutes.
 - c. Carefully sand or file the porous plug junction.

APPENDIX G-5

EM SURVEY USING THE GEONICS EM-31-D



Technical Note TN-6

**ELECTROMAGNETIC TERRAIN
CONDUCTIVITY MEASUREMENT
AT LOW INDUCTION NUMBERS**



GEONICS LIMITED

Leaders in Electromagnetics



GEONICS LIMITED

1745 Meyerside Drive, Mississauga, Ontario, Canada L5T 1C5 Tel. (416) 676-9580, Telex 06-968688, Cables: Geonics

Technical Note TN-6

**ELECTROMAGNETIC TERRAIN
CONDUCTIVITY MEASUREMENT
at
LOW INDUCTION NUMBERS**

JD McNEILL

October, 1980

I. INTRODUCTION

The measurement of terrain resistivity to map geology has been utilized for over half a century. Several shortcomings, however, have prevented this technique from being widely accepted for engineering purposes. The first of these is that conventional galvanic resistivity surveys require a relatively large amount of manpower to execute and are thus expensive. Secondly, the actual value of resistivity itself is seldom diagnostic; it is the lateral or vertical variations of resistivity which form the basis of any interpretation. However the high cost of resistivity surveying generally means that fewer measurements are made than would be desirable, with the result that either (i) the survey area is not made large enough to establish a reasonable background against which the anomalous areas are to be delineated or (ii) the anomalous area itself is obscure and lacks definition.

An additional problem inherent to conventional resistivity techniques is that although the effective depth of exploration is determined by the selected inter-electrode spacing, resistive inhomogeneities which are small compared to this depth but which are located near the potential electrodes can cause a significant error in the measurement. Such fluctuations in the measured results are truly geological "noise" because it is not possible to determine the physical size, resistivity contrast, or location of the source. As a result of such inhomogeneities resistivity profiles carried out at constant interelectrode spacing tend to be noisy, limiting the resolution in resistivity that can be achieved, even though the instrumentation itself is capable of producing much higher accuracy.

It was an awareness of both the advantages of resistivity for engineering geophysical surveys and the disadvantages of conventional resistivity techniques that led Geonics Limited to examine the possibility of employing electromagnetic (inductive) techniques as an alternative for resistivity surveys. With the development of the EM31 and the EM34-3 it is now possible to map terrain conductivity virtually as fast as the operator(s) can walk; furthermore the sample volume is averaged in such a manner as to yield unexcelled resolution in conductivity.

These patented instruments have been designed to cover the range of depths generally useful for engineering geophysics; the EM31, one-man portable, has an effective depth of approximately 6 meters and the EM34-3, two-man portable, has stepwise selectable depths from 7.5 meters to a maximum of 60 meters.

Typical applications for the EM31 and EM34-3 instrumentation are:

- (i) Delineating regions of permafrost (frozen pore water)
- (ii) Locating gravel
- (iii) Extending known gravel deposits
- (iv) Mapping saline intrusions
- (v) Detecting cavities in carbonate rocks
- (vi) Mapping pollution plumes in groundwater
- (vii) Mapped bedrock topography
- (viii) Mapping terrain conductivity for electrical grounding
- (ix) General geological mapping (soil types, fault and fracture zones, etc.)
- (x) Archaeological exploration
- (xi) Locating pipes (EM31) and metallic-type conductors

This technical note describes both the principles and the instrumentation employed to measure terrain conductivity using electromagnetic techniques at low induction numbers. For a detailed discussion of the concept of terrain resistivity/conductivity and of the various factors that control this parameter the reader is referred to Geonics Limited Technical Note "Electrical Conductivity of Soils and Rocks".

II. PRINCIPLE OF OPERATION

The application of electromagnetic techniques to the measurement of terrain resistivity, or more properly, conductivity* is not

*Conductivity is preferred with inductive techniques since the response is generally proportional to conductivity and inversely proportional to resistivity.

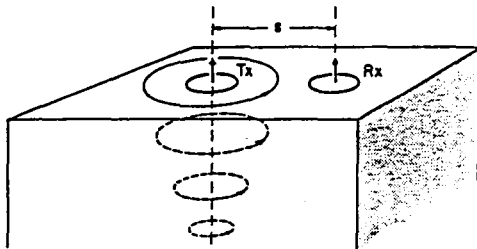


FIGURE 1. Induced current flow (homogeneous halfspace).

new and excellent descriptions of this technique are given in the literature [1], [2].

Consider Figure 1 in which a transmitter coil Tx energized with an alternating current at an audio frequency, is placed on the earth (assumed uniform) and a receiver coil Rx is located a short distance s away. The time-varying magnetic field arising from the alternating current in the transmitter coil induces very small currents in the earth. These currents generate a secondary magnetic field H_s which is sensed, together with the primary field, H_p , by the receiver coil.

In general this secondary magnetic field is a complicated function of the intercoil spacing s , the operating frequency, f , and the ground conductivity σ . Under certain constraints, technically defined as "operation at low values of induction number" (and discussed in detail in the appendix) the secondary magnetic field is a very simple function of these variables. These constraints are incorporated in the design of the EM31 and EM34-3 whence the secondary magnetic field is shown to be:

$$\frac{H_s}{H_p} = \frac{i\omega\mu_0\sigma s^2}{4} \quad (1)$$

where H_s = secondary magnetic field at the receiver coil

H_p = primary magnetic field at the receiver coil

$\omega = 2\pi f$

f = frequency (Hz)

μ_0 = permeability of free space

σ = ground conductivity (mho/m)

s = intercoil spacing (m)

$i = \sqrt{-1}$

The ratio of the secondary to the primary magnetic field is now linearly proportional to the terrain conductivity, a fact which makes it possible to construct a direct-reading, linear terrain conductivity meter by simply measuring this ratio. Given H_s/H_p , the apparent conductivity indicated by the instrument is defined from equation (1) as

$$\sigma_a = \frac{4}{\omega\mu_0 s^2} \left(\frac{H_s}{H_p} \right) \quad (2)$$

The MKS units of conductivity are the mho (Siemen) per meter or, more conveniently, the millimho per meter.

III. INSTRUMENTATION

The EM31 (shown in Figure 2) has an intercoil spacing of 3.7 meters, which yields an effective depth of exploration of about 6 meters. The instrument can also be operated on its side, in which



FIGURE 2. EM31 in field operation.

case as will be seen in Section IV., the effective depth of exploration is reduced to approximately 3 meters. The instrument is one-man portable and can be used either in "station-by-station" mode or read continuously. The presence of layering in the earth can be detected by raising the instrument and noting the readings as a function of instrument height. If the earth is two-layered the conductivity of both layers and the upper layer thickness can be resolved.

The EM34-3 which is two-man portable has the two coils flexibly connected (Figure 3). The intercoil spacing is measured electronically so that the receiver operator simply reads a meter to accurately set the coils to the correct spacing, which can be 10, 20, or 40 meters so as to directly vary the effective depth of exploration as shown in Table I.



FIGURE 3. EM34-3 in field operation.

TABLE I. Exploration depths for EM34-3 at various intercoil spacings

Intercoil Spacing (meters)	Exploration Depth (meters)	
	Horizontal Dipoles	Vertical Dipoles
10	7.5	15
20	15	30
40	30	60

To measure terrain conductivity the transmitter operator stops at the measurement station; the receiver operator moves the receiving coil backwards or forwards until his meter indicates correct intercoil spacing and he reads the terrain conductivity from a second meter. The procedure takes 10 to 20 seconds. The coils are normally carried with their planes vertical (horizontal dipole mode) since at this configuration the measurement is relatively insensitive to misalignment of the coils. In the event that the greater depth of penetration resulting when the two coils are in the vertical dipole mode is desired, more care must be taken with intercoil alignment. Because of the relatively short intercoil spacing correct alignment is usually not difficult to achieve.

Both instruments are calibrated to read terrain conductivity in millimhos per meter. To convert these readings to resistivity in ohmmeters one simply divides them into 1,000, i.e. 50 millimhos per meter is the equivalent of 20 ohmmeters.

IV. SURVEY TECHNIQUES AND INTERPRETATION

For either the EM31 or EM34-3 it can be shown that in a homogeneous or horizontally stratified earth the current flow is entirely horizontal. Furthermore under the constraints by which the instruments are designed the current flow at any point in the ground is independent of the current flow at any other point since the magnetic coupling between all current loops is negligible. Finally, under these constraints the depth of penetration is limited only by the intercoil spacing. We say that the depth of penetration is "source" or "geometry" limited rather than "skin depth" limited since it is now controlled by the fall-off with distance of the dipolar transmitter field. For this reason all dimensions are normalized with respect to the intercoil spacing in subsequent sections of this technical note.

IV. 1. Instrumental Response as a Function of Depth (Homogeneous Halfspace)

Consider a homogeneous halfspace on the surface of which is located an EM31 or an EM34-3 transmitter as shown in Figure 4. Fixing our attention on a thin layer of thickness dz at depth z (where z is the depth divided by the intercoil spacing s) it is possible to calculate the secondary magnetic field in the receiver coil arising from all of the current flow within this or any other horizontal thin layer. One can thus construct the function $\phi_v(z)$ shown in Figure 4 which describes the relative contribution to the secondary magnetic field arising from a thin layer at any depth z . We see from this figure that material located at a depth of approximately $0.4s$ gives maximum contribution to the secondary magnetic field but that material at a depth of $1.5s$ still contributes significantly. It is interesting to note that the ground at zero depth, i.e. the near surface material,

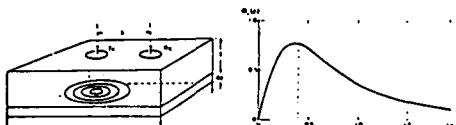


FIGURE 4. Relative response versus depth for vertical dipoles. $\phi_v(z)$ is the relative contribution to H_z from material in a thin layer dz located at (normalized) depth z .

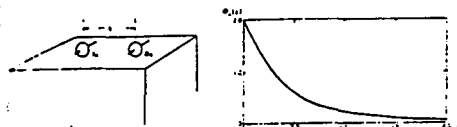


FIGURE 5. Relative response versus depth for horizontal dipoles

and a very small contribution to the secondary magnetic field and therefore this coil configuration is insensitive to changes in near surface conductivity.

Figure 5 illustrates the function of Figure 4 for the case of both transmitter and receiver dipoles horizontal coplanar rather than vertical coplanar. For the coil configuration of Figure 5 (commonly used for the EM34-3 since it is less critical to intercoil alignment) the relative contribution from material near-surface is large and the response falls off monotonically with depth.

A comparison of the function ϕ for both coil configurations in Figure 6 emphasizes the different manner in which they respond to material at different depths. The difference is important since either instrument can be rolled over so that the vertical dipole transmitter/receiver geometry becomes a horizontal dipole transmitter/receiver geometry and vice versa. As will be seen later, this feature is useful in diagnosing and defining a layered earth. The figure also shows that for regions greater than one intercoil spacing in depth the vertical transmitter/receiver dipole gives approximately twice the relative contribution of the horizontal transmitter/receiver dipole.

To summarize, with either horizontal or vertical transmitter/receiver dipole orientation it is possible to construct a function which gives the relative response to the secondary magnetic field at the receiver from a thin layer of ground at any depth. That this is possible arises from the fact that (i) all current flow is horizontal and (ii) all current loops are independent of all other current loops. It should be noted that it is not possible to construct such functions for conventional resistivity techniques.

Finally, since as shown in Section II the definition of apparent conductivity is given in terms of the secondary magnetic field at the receiver, the functions in Figure 6 also give the relative contribution

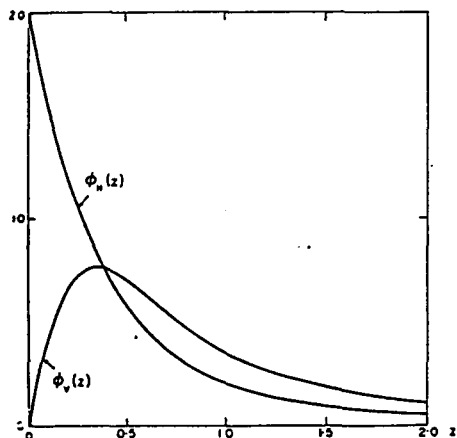


FIGURE 6. Comparison of relative responses for vertical and horizontal dipoles.

from material at different depths to the *apparent conductivity* indicated by the instrument meter. The integral of either function from zero to infinity gives the total secondary magnetic field at the receiver coil from a homogeneous halfspace which is directly related to the electrical conductivity of the halfspace by equation (1). It is therefore possible to state with great precision the relative influence of material at different depths to the indicated apparent conductivity.

IV. 2. Multi-Layered Earth Response

The functions shown in Figure 6 are useful for describing the relative sensitivity of either of the two coil configurations to material at various depths. However a function derived from them is more useful for performing calculations. It is defined as the relative contribution to the secondary magnetic field or apparent conductivity from all material below a depth z and is given by

$$R_v(z) = \int_z^\infty \phi_v(z) dz \quad (3)$$

Called the cumulative response, this function is illustrated in Figure 7 for vertical coplanar transmitter/receiver dipoles. The figure shows, for example, that for this configuration all material below a depth of two intercoil spacings yields a relative contribution of approximately 0.25 (i.e. 25%) to the secondary magnetic field at the receiver coil.

Suppose now that our homogeneous halfspace has a conductivity of 20 millimhos per meter (50 ohmmeters). The equipment having been calibrated according to equation (2), the output meter indicates 20 millimhos per meter. From Figure 7 we observed that the material below two intercoil spacings contributed 25% to the secondary magnetic field and therefore 25% to the indicated meter reading. Suppose that we replace this deep material with an infinitely resistive (zero conductivity) substance. Since we have reduced to zero the 25% that this material contributed to the meter reading the new reading will be 75% of 20, or 15 millimhos per meter. Conversely, if we leave all of the material below two intercoil spacings at 20

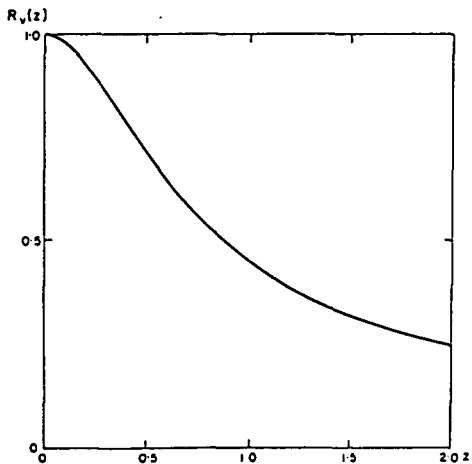


FIGURE 7. Cumulative response versus depth for vertical dipoles. $R_v(z)$ is the relative contribution to H_z from all material below a (normalized) depth z .

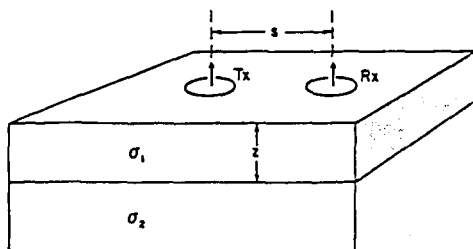


FIGURE 8. Two layer earth model.

millimhos per meter but make all material above two intercoil spacings infinitely resistive the meter reading will fall from the original 20 millimhos per meter for the homogeneous half space to 5 millimhos per meter, since, if all of the material below two intercoil spacings contributed 25% of the meter reading, all of the material above two intercoil spacings must contribute 75%; when removed the meter reading becomes 0.25×20 or 5 millimhos per meter.

From this example we see that there is a simple way to calculate the instrument reading on an arbitrarily layered earth as long as the intercoil spacing is much less than the skin depth in all of the layers. We simply add the contribution from each layer independently, weighted according to its conductivity and depth according to Figure 7. For example assume that we have a two-layer case as shown in Figure 8. The contribution from the upper layer is given by

$$\sigma_u = \sigma_1 [1 - R_V(z)] \quad (4a)$$

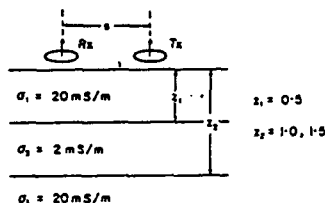
since all of the material below zero depth yields a relative contribution of unity or 100% to the meter reading. Conversely all of the material in the lower layer adds a contribution given by

$$\sigma_b = \sigma_2 R_V(z) \quad (4b)$$

and the actual instrument reading will therefore be the sum of these two quantities

$$\sigma_s = \sigma_1 [1 - R_V(z)] + \sigma_2 R_V(z) \quad (5)$$

If the earth is three-layered as shown in Figure 9 the same procedure is employed to determine the instrumental response. In this example the calculations are performed for different middle layer thicknesses.



$$\sigma_s = \sigma_1 [1 - R(z_1)] + \sigma_2 [R(z_1) - R(z_2)] + \sigma_3 R(z_2)$$

$$z_1 = 0.5, \sigma_s = 20 [1 - 0.70] + 2 [0.70 - 0.44] + 20 \times 0.44 = 15.3 \text{ mmo/m}$$

$$z_1 = 1.5, \sigma_s = 20 [1 - 0.70] + 2 [0.70 - 0.32] + 20 \times 0.32 = 13.2 \text{ mmo/m}$$

FIGURE 9. Calculation of response to three layer earth - center layer thickness varying.

The ease with which such calculations are performed facilitates survey preparation and interpretation. It is sometimes possible to make advance estimates of the electrical properties of the material to be encountered during a survey or, alternatively, once the operator can obtain the same information from sample measurements of the different materials. The procedures outlined above are then employed to estimate the apparent conductivity measured under various terrain conditions. Examples of such calculations for the EM31 are shown in Figure 10. As is seen in the appendix, the algebraic expressions for $\phi(z)$ and $R(z)$ are very simple and can easily be programmed on hand held calculators.

In Figure 10 the vertical dimensions are greatly exaggerated with respect to the horizontal dimensions. The question arises as to what degree of lateral uniformity is required before the earth can be considered as horizontally stratified or homogeneous. Survey experience indicates that if the ground conductivity does not significantly vary with horizontal distance within a radius of one intercoil spacing from the instrument the ground can be considered to be laterally uniform.

The above discussion referred to the use of vertical transmitter/receiver dipoles; it is equally possible to construct a cumulative response function for the horizontal coplanar dipole configurations and Figure 11 illustrates this function for both coil configurations. A comparison of the two curves illustrates that the vertical dipole mode of operation has approximately twice the effective exploration depth of the horizontal dipole mode.

IV. 3. Comparison with Conventional Resistivity Techniques

Many readers will be familiar with the two-layer curves employed to interpret data from conventional resistivity surveys using a Wenner array of four equally spaced electrodes. Using the technique described in the previous section it is a simple matter to calculate two-layer curves for the electromagnetic technique; Figure 12 shows such curves for both the vertical and horizontal dipole configurations superimposed on standard Wenner curves. The general shape is similar but there are marked differences in detail. For vertical coplanar transmitter/receiver dipoles we see that when the substrate is the more resistive the response of the two systems is similar; however when the substrate is the more conductive the electromagnetic technique sees deeper in that the influence of the substrate, for a given conductivity contrast, is felt at smaller intercoil spacing than inter-electrode spacing. This is a general characteristic of electromagnetic systems which prefer to look through an insulator to a conductor rather than through a conductor to an insulator.

For the horizontal dipole configuration if the lower layer is the more resistive the effective exploration depth of the inductive technique is slightly less than the Wenner array; however, once again, in the case where the lower layer is the more conductive the exploration depth of the inductive technique is substantially greater.

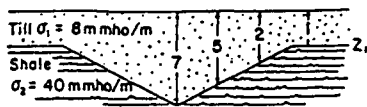
IV. 4. Resolution of Two-Layered Earth by Varying Lateral Spacing

The principal advantage of the inductive electromagnetic technique over conventional resistivity lies in the speed and accuracy with which lateral changes of terrain conductivity can be measured. However this technique can also be used to measure the vertical variation of conductivity by expanding the intercoil spacing in a manner analogous to that in which the electrode spacing is expanded in conventional resistivity sounding techniques. The current state-of-the-art, however, is such that relatively few intercoil spacings can be employed; for example the EM31 can be operated with an intercoil spacing of 10, 20 or 40 meters. This feature is somewhat mitigated by the fact that the instruments can be used in either the vertical or horizontal dipole modes which, as shown in a previous section, exhibit different sensitivity to various depths thus yielding more information than would be available by simply using three spacings with one coil orientation.

To interpret a two-layer geometry the two-layer curves for both dipole configurations are superimposed on a common plot as shown

CROSS-SECTIONS

BURIED RIVER VALLEY



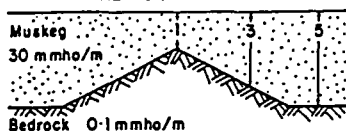
$$\frac{\sigma_0}{\sigma_1} = 1 - R(Z_1) + k_2 R(Z_1)$$

$$k_2 = \frac{\sigma_2}{\sigma_1} = \frac{40}{8} = 5$$

$Z_1(m), \sigma_0(mmho/m)$

1	32.6
2	26.9
5	18.6
7	16.0

BEDROCK HIGH



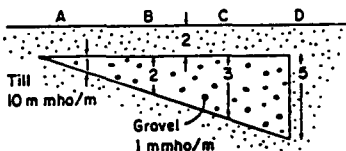
$$\frac{\sigma_0}{\sigma_1} = 1 - R(Z_1) + k_2 R(Z_1)$$

$$k_2 = \frac{\sigma_2}{\sigma_1} = \frac{0.1}{30} = 0.0033$$

$Z_1(m), \sigma_0(mmho/m)$

1	6.9
3	15.9
5	20.1

GRAVEL DEPOSIT



$$\frac{\sigma_0}{\sigma_1} = 1 - R(Z_1) + k_2 [R(Z_1) - R(Z_2)] + k_3 R(Z_2)$$

$$k_2 = \frac{\sigma_2}{\sigma_1} = \frac{1}{10} = 0.10$$

$$k_3 = \frac{\sigma_3}{\sigma_1} = 1.00$$

station $\sigma_0(m mho/m)$

A	8.9
B	8.2
C	7.7
D	6.9

FIGURE 10. EM31 calculated response across various geological features, using $R(Z)$ corrected for instrument operation at waist (1 meter) height. Intercoil separation $s = 3.67$ meters.

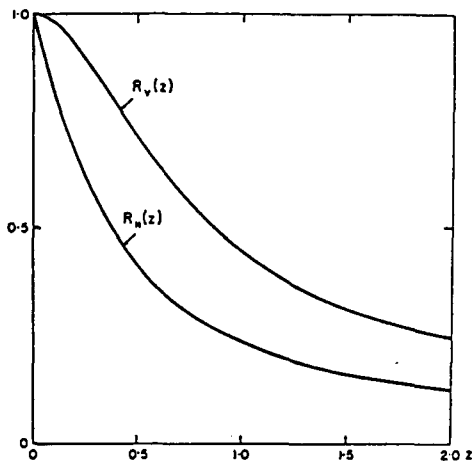


FIGURE 11. Cumulative response versus depth for vertical and horizontal dipoles.

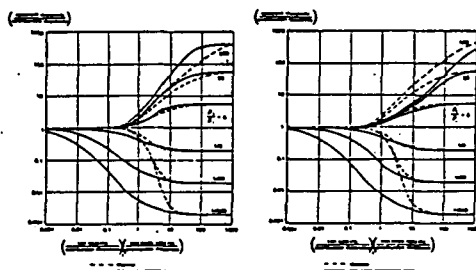


FIGURE 12. Comparison of Wenner array and inductive electromagnetic sounding curves for a two layer earth.

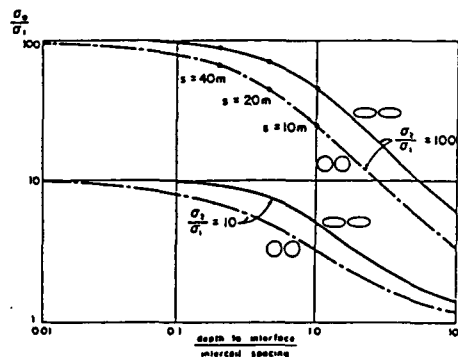


FIGURE 13. Two layer earth response curves ($\sigma_2/\sigma_1 = 10, 100$; intercoil spacing varied). Dots indicate typical survey results.

in Figure 13. The six data points obtained by making measurements with two coil orientations and three intercoil spacings are plotted to the same scale on a piece of transparent paper and are translated vertically and horizontally on the two-layer curves to ascertain whether a satisfactory fit can be achieved. In the event that such a fit can be made, the earth does exhibit two-layer characteristics and the values of conductivity for both layers and the thickness of the upper layer are directly read off.

IV. 5. Resolution of Two-Layered Earth by Varying Instrument Height

In the case of the EM31 the intercoil spacing is rigidly fixed so that the technique described above is not available to analyse a layered earth. It is, however, possible to raise the instrument above the ground, measuring the apparent conductivity as a function of instrument height for both the vertical and horizontal dipole configurations. This has the effect of shifting the response curves of Figure 6 upwards through the various regions of the earth and the variation of apparent conductivity with height is therefore of diagnostic value in determining the nature of any layering. It is a straightforward matter to calculate the response of the instrument as a function of height for various two-layered earth geometries and typical curves are shown in Figure 14b. To use the curves one simply plots the measured apparent conductivity versus height for both coil configurations on a piece of transparent paper to the same scale as Figure

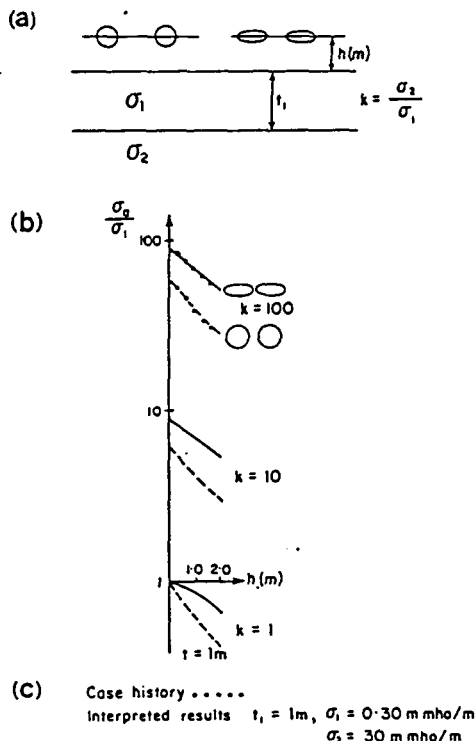


FIGURE 14. Two layer earth response curves ($\sigma_2/\sigma_1 = 1, 10, 100$; instrument height varied). Dots are actual survey results.

14b and shifts the plotted data vertically until good agreement is achieved with one of the curves, whereupon the two conductivities and the upper layer thickness are immediately determined as in the illustrated case history of Figure 14c.

In the event that the conductivity of either one of the two layers is known to be much less than the other, so that its contribution to the meter reading is negligible, it is simply necessary to lay the instrument on the ground, take a reading, lay it on its side, take a second reading, and from these two values one can immediately calculate the conductivity of the more conductive layer and the thickness of the upper layer.

V. ADVANTAGES AND DISADVANTAGES OF INDUCTIVE TERRAIN CONDUCTIVITY MEASUREMENTS

V. 1. Advantages

The advantages of the use of inductive electromagnetic techniques to measure terrain conductivity are as follows:

- Excellent resolution in conductivity.* It was stated in Section I that a problem with conventional resistivity was that the presence of localized resistivity inhomogeneities near the potential electrodes caused large errors. If we examine the current flow in a homogeneous halfspace for the inductive technique described herein we realize that in the vicinity of the transmitter the current density is very high and we might expect the presence of a conductive inhomogeneity located here to have a large effect. However where the current density is high, the radius of the current loops is small and their distance from the receiver coil large, so that these loops do not couple well magnetically with the receiver. The effect of changing this current by varying the local conductivity is consequently negligible. The lateral extent of the volume of earth whose conductivity is sensed by the inductive technique is approximately the same as the vertical depth. The result is that small changes in conductivity, for example of the order of 5% or 10%, are easily and accurately measured.
- No current injection problems.* Since currents are magnetically induced in the earth, current injection problems encountered with conventional resistivity in materials such as gravel, bedrock, permafrost, snow and ice, etc., are not encountered with this type of instrumentation.
- Simple multi-layered earth calculations.* This matter is dealt with at length in Section IV.
- Easy, rapid measurements.* A problem with the conventional Wenner array is that in order to survey to an effective depth a the array must be $3a$ in length and the total length of wire required $4a$, used in four sections. This presents many opportunities for snagging and breaking the wire. Furthermore each measurement requires insertion of four electrodes and relatively careful measurement of the inter-electrode spacing. These features are avoided with the inductive electromagnetic techniques and it is no exaggeration to say that a survey can often be carried out five to ten times faster using this technique. Indeed with either the EM31 or the EM34-3 it is usually possible under average terrain conditions to survey 5' to 7 line-kilometers a day with a station spacing of 25 or 50 meters.

V. 2. Disadvantages

As with all geophysical instruments, there are some limitations and disadvantages to the use of inductive electromagnetic techniques and these are as follows:

- Limited dynamic range (1 - 1000 m mhos per meter).* At low values of terrain conductivity it becomes difficult to magnetically induce sufficient current in the ground to produce a detectable magnetic field at the receiver coil. Conversely at high values of conductivity the quadrature component of the received magnetic field is no longer linearly proportional to terrain conductivity as is shown in the appendix.
- Setting and maintaining the instrument zero.* Ideally in order to set the zero the instrument would be suspended in free space

and the zero set there. The more acceptable alternative is to search out a region of very resistive ground, to accurately measure its conductivity using conventional techniques, and to set the instrumental zero at that location. This is the procedure which is actually followed.

It is necessary that this zero be accurately maintained over long periods of time and over the wide variations of temperature encountered during geophysical survey in various parts of the world. This produces tight constraints on the circuitry, with the result that the zero may be in error by up to ± 0.2 mmhos per meter. Such an error would be negligible over the usual range of terrain conductivities; however in the event that measurements are being made on highly resistive ground the zero error can become significant.

- (iii) **Limited Vertical Sounding Capability.** In theory it is possible to use a system such as the EM34-3 at a continuum of intercoil spacings to yield more information about electrical layering in the ground. To achieve a wide variety of inter-electrode spacings, with conventional resistivity equipment is simple; in the case of the inductive electromagnetic technique the rapid fall-off of the magnetic field from the dipole transmitter introduces a serious dynamic range problem. In due course there will undoubtedly be instrumentation with a wider variety of spacings at the expense of additional complexity.

VII. CASE HISTORIES

This section describes several case histories obtained with the EM31 and the EM34. The surveys (i) illustrate the resolution in conductivity that can be achieved, (ii) compare the results obtained with conventional resistivity and (iii) illustrate the use of the latter for locating sand, gravel and conductive minerals, determining bedrock topography (including locating a buried river channel) and mapping the pollution plume from a land-fill site. In some cases the indicated conductivity has been converted to resistivity to facilitate comparison with conventional resistivity survey results.

Case History #1

Location: Mississauga, Ontario
Instrument: EM31

Application: Illustrates resolution and repeatability of EM31

For this case history a Rustrak chart recorder was used to monitor the output of an EM31. A line of length 200 meters was traversed in a heil in both easterly and westerly directions. Figure 15 demon-

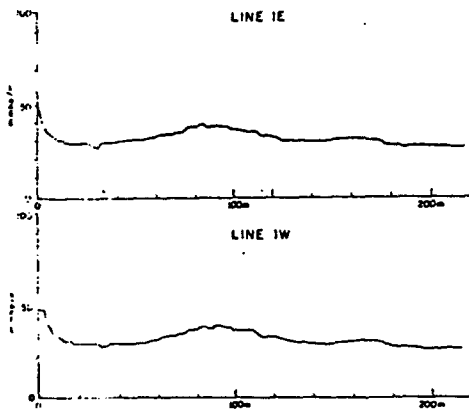


FIGURE 15.

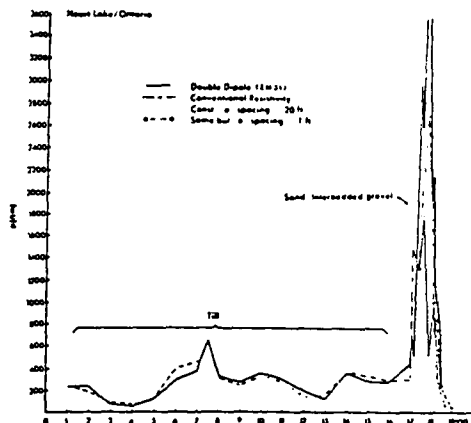


FIGURE 16. Test survey line - Heart Lake, Ont.

strates that the instrument is resolving conductivity changes of less than 1 mmhos/m (1% of full scale deflection) and that the repeatability is of the same order. In fact the repeatability is limited in this case by the resolving power of the chart recorder itself. It should furthermore be noted that the instrument is detecting spatial changes in conductivity of a few meters in length - compatible with the intercoil spacing of 3.7 meters.

Case History #2

Location: Heart Lake, Ontario
Instruments: EM31
Conventional resistivity apparatus
Application: Location of sand/gravel
Comparison of EM31 and conventional resistivity

In this survey a line 1900 ft. (580 meters) in length was surveyed with a measurement interval of 100 ft. (30 meters). The survey area was generally located on a buried esker, however the last few survey stations, 17 + 00 to 19 + 00, traversed a region of exposed sand and gravel (often occurring in the form of concretions) and over this portion of the line measurements were made every 10 ft. (3.0 meters).

The conventional resistivity profile was carried out using a Wenner array with an spacing of 20 ft. (6.1 meters) except between stations 17 + 00 and 19 + 00 where the spacing was reduced to 1 ft. (0.30 meters).

In general the correlation between the two sets of data is excellent, and demonstrates the ability of the EM31 to generate good quantitative data even in regions of low conductivity. Over the esker the EM31 was actually read continuously down the line - the data was recorded only at the 100 ft. intervals, with the exception of the reading at station 7 + 50 which was also recorded since it was noted that a conductivity low occurred there. Such an anomaly was, of course, missed by the conventional resistivity where measurements were only made every 100 ft.

Both sets of data become rather erratic between stations 17 + 00 and 19 + 00 as a result of the very rapid lateral changes in resistivity arising from the concreted material referred to above.

Case History #3

Location: Cavendish, Ontario.
Instrument: EM31
Application: Location of metallic type conductors

This survey line, of length 2000 ft. (610 meters), is located at a site

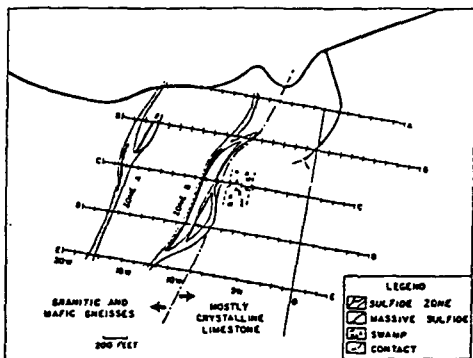


FIGURE 17. Geologic map of the Cavendish test site and the grid of traverse lines used in geophysical studies (after Ward et al [3]).

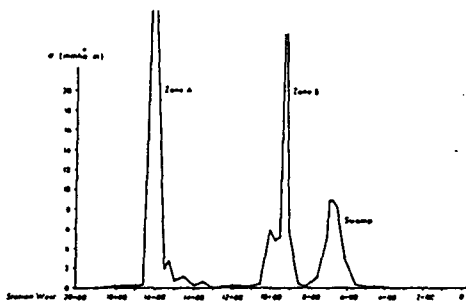


FIGURE 18. EM31 survey of Cavendish test range Line 'C'.

in Ontario which is often used by Canadian instrumentation manufacturers to test new electromagnetic geophysical equipment. The survey, along line C, illustrates response from both the swamp and the two zones of metallic mineralization. Although measurements were only taken every 50 ft. (15 meters) both zones are well delineated and when such high responses are encountered localization to within a few meters is quickly and easily carried out.

Inasmuch as the EM31 and EM34-3 were designed to map terrain conductivity at the conductivity levels encountered in typical soils both instruments are extremely sensitive electromagnetic detectors. For example on the most sensitive scale, full scale deflection for the EM31 is 800 ppm of the primary magnetic field and for the EM34-3 it is 3800 ppm. Such sensitivity makes either instrument useful for detecting metallic type conductors at what are very low conductivity levels by normal standards.

Case History #4

Location: Mississauga, Ontario
Instruments: EM31, EM34
Application: Determination of bedrock topography

Total line length for this survey was 8400 ft. (2600 meters) and measurements were made every 100 ft. (30 meters) with both the EM31 and the EM34 - an earlier version of the EM34-3 which had two intercoil spacings vis. 100 ft. (30 meters) and 50 ft. (15 meters). The survey was performed to outline the cross-sectional profile of a

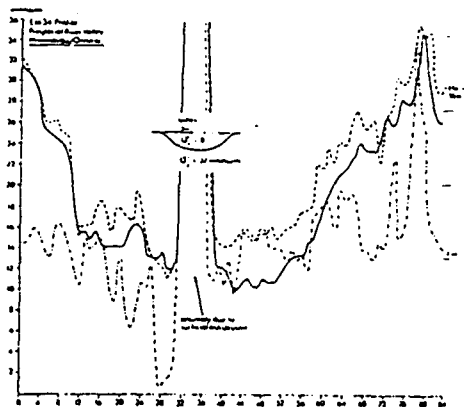


FIGURE 19. EM31 and EM34 survey line over preglacial river valley, Mississauga, Ontario.

buried preglacial river valley whose existence had been suggested from water-well data. At either intercoil spacing the time required for the EM34 profile was 1-1/2 hours, resulting in approximately 30 survey measurement per minute - including the time to walk the feet between measurement stations. The time taken for the subsequent EM31 survey was similar.

Typical bedrock conductivity in the area is approximately mmo/m, whereas an average value for the conductivity of infilling glacial till is of the order of 8 to 12 mmo/m. Thus the EM31 at either intercoil spacing yields approximately 30 mmo/m at valley edges where the overburden is thin and 12 to 14 mmo/m at the valley centre. The EM34 yields values of 14 to 18 mmo/m at valley edges (slightly affected by the presence of bedrock), approximately 10 mmo/m at the valley centre. The interdepth of the valley, based on the model shown in the figure, is approximately 120 feet (36 meters) which is in reasonable agreement with the water-well data value of 150 feet (45 meters), bearing in mind that the three sets of data show that a two-layer model over simplification.

The conductivity high which occurs between stations 32 and 33 results from a very large pile of waste furnace ash lying on the surface.

Case History #5

Location: Camp Borden, Ontario
Instruments: EM31, EM34
Application: Mapping groundwater salinity
Comparison of EM34 and conventional resistivity

Geophysical surveys were carried out over a sanitary landfill, using, in addition to other instruments, an EM31, EM34 and conventional resistivity [4]. The survey results in the accompanying figures illustrate the good agreement between these techniques also indicate the reduction in survey time achieved using induced electromagnetic techniques. Particularly interesting are the vertical variations in resistivity as shown by the EM31 at 3.7 m intercoil spacing and the EM34 at 15 and 30 m spacing.

VII. SUMMARY

This technical note describes in detail the principles of measuring the electrical conductivity of the ground using magnetically induced

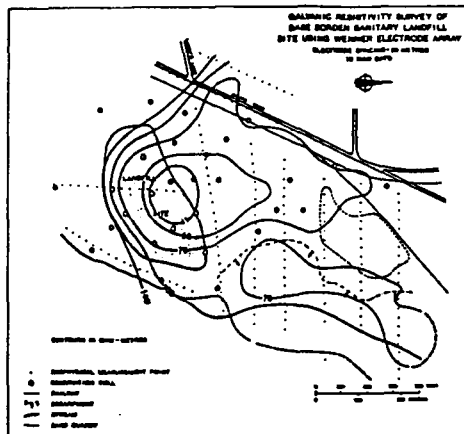


FIGURE 20(a).

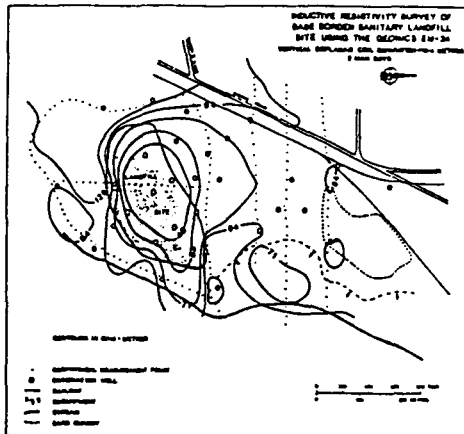


FIGURE 20(c).

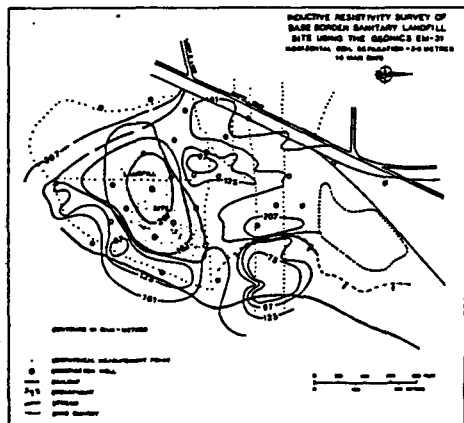


FIGURE 20(b).

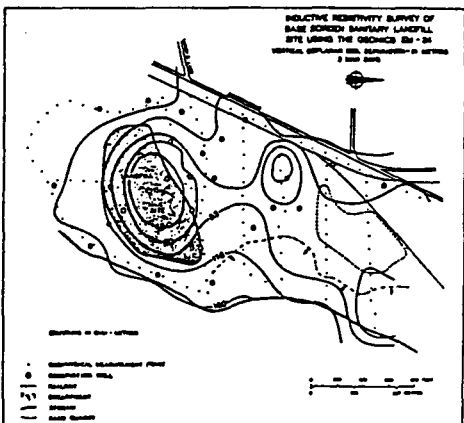


FIGURE 20(d).

currents at low frequencies. It has been shown that certain advantages can be derived from working at low values of induction number. Amongst these are excellent resolution in conductivity, a substantial reduction in man-hours necessary to carry out a conductivity survey and a simplification in the calculation of layered earth response.

Two points should be kept constantly in mind when performing surveys of this type to map geology. The first is that these instruments map only the electrical conductivity. If the conductivity does not vary significantly with the geological environment, or if parameters other than the geology also influence the conductivity, the survey results may be difficult to interpret.

The second point is that measurement of terrain conductivity, like any other geophysical measurement, must begin and end with geology. Such measurements are only an aid to help visualize geological conditions which cannot be seen. It is always necessary to interpret

geophysical data against known geology from out-crops, boreholes, or any other such "bench marks". Geophysical measurements can be very effective by allowing interpolation between such sources, or extrapolation away from them. However in every case knowledge derived from geophysical measurements must be eventually re-confirmed against known geological conditions.

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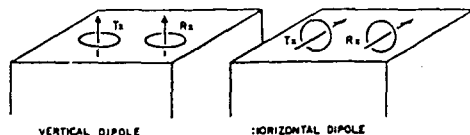


FIGURE A1. Vertical and horizontal dipole coil configurations.

APPENDIX: Theory of Operation at Low Induction Numbers

Consider the two coil configurations shown in Figure A1. In each case the transmitter coil is energized with alternating current at a frequency f Hertz. The measured quantity is the ratio of the secondary magnetic field H_s at the receiver when both coils are lying on the surface of the homogeneous half-space of conductivity σ to the primary magnetic field H_p in the absence of the half-space (i.e. as if the coils were in free space). The spacing between the coils is s meters.

The field ratios for vertical and horizontal dipole configurations are given by equations (1) and (2) respectively.

$$\left(\frac{H_s}{H_p}\right)_v = \frac{2}{(\gamma s)^2} [9 - [9 + 9\gamma s + 4(\gamma s)^2 + (\gamma s)^3] e^{-\gamma s}] \quad (1)$$

$$\left(\frac{H_s}{H_p}\right)_h = 2 \left[1 - \frac{3}{(\gamma s)^2} + [3 + 3\gamma s + (\gamma s)^2] \frac{e^{-\gamma s}}{(\gamma s)^2} \right] \quad (2)$$

$$\text{where } \gamma = \sqrt{i\omega\mu_0\sigma}$$

$$\omega = 2\pi f$$

$$f = \text{frequency (Hz)}$$

$$\mu_0 = \text{permeability of free space}$$

$$i = \sqrt{-1}$$

These expressions are complicated functions of the variable γs which is in turn a reasonably complicated (complex) function of frequency and conductivity. However, as will be shown below, under certain conditions they can be greatly simplified.

A well known characteristic of a homogeneous half-space is the electrical skin depth δ , which is defined as the distance in the half-space that a propagating plane wave has travelled when its amplitude has been attenuated to $1/e$ of the amplitude at the surface. The skin depth is given by

$$\delta = \sqrt{\frac{2}{\omega\mu_0\sigma}} = \frac{\sqrt{2i}}{\gamma} \quad (3)$$

and therefore

$$\gamma s = \sqrt{2i} \frac{s}{\delta} \quad (4)$$

The ratio s/δ , the intercoil spacing divided by the skin depth, is defined as the induction number B , whereupon

$$\gamma s = \sqrt{2i} B \quad (5)$$

Now if B is much less than unity (ie $\gamma s \ll 1$) it is a simple matter to show that the field ratios of equations (1) and (2) reduce to the simple expression

$$\left(\frac{H_s}{H_p}\right)_v \approx \left(\frac{H_s}{H_p}\right)_h \approx \frac{iB^2}{2} = \frac{i\omega\mu_0\sigma s^2}{4} \quad (6)$$

which is the equation given in Section II.

The magnitude of the secondary magnetic field is now directly proportional to the ground conductivity and the phase of the secondary magnetic field leads the primary magnetic field by 90° .

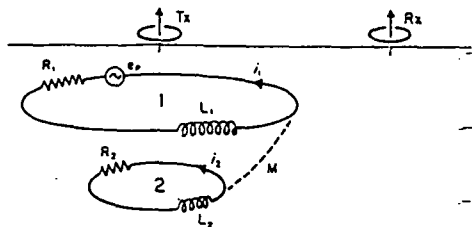


FIGURE A2. Electrical model for vertical dipoles.

To make B much less than unity we see that we must make s very much less than δ and thus

$$\omega \ll \frac{2}{\mu_0\sigma s^2}$$

That is, having decided on a value for s (which fixes the effective depth of penetration under the condition $B \ll 1$), the maximum probable ground conductivity is estimated and the operating frequency is chosen so that equation (7) is always satisfied.

The apparent conductivity which the instrument reads is then defined by

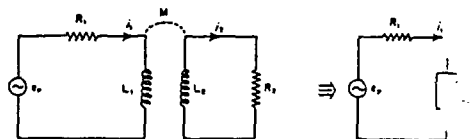
$$\sigma_a = \frac{4}{\omega\mu_0 s^2} \left(\frac{H_s}{H_p}\right)_{\text{quadrature component}} \quad (8)$$

To examine the reasons for this simplification let us focus attention on the vertical dipole coil configuration shown in Figure A2. All since symmetry makes this configuration the simplest to understand.

Consider current loop 1. The primary emf e_s causing this current to flow is given (through Faraday's law) by the time rate of change of the primary magnetic flux from the transmitter through this loop. Three impedances cause the current to be limited. These arise from (i) the electrical resistance R_1 of the loop, (ii) the fact that the current i_1 generates its own magnetic field which causes a time-varying secondary magnetic flux through the loop (self-inductance, L_1), and (iii) the fact that all other current loops such as i_2 generate their own magnetic fields which in turn cause a time-varying magnetic flux to link with loop 1 (mutual inductance, M).

The equivalent circuit for this configuration is easily derived from elementary circuit theory with the result shown in Figure A3.

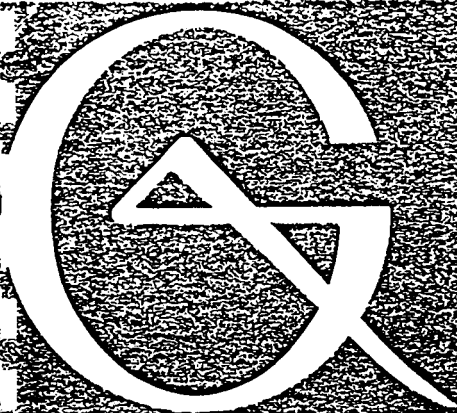
The complex impedance Z incorporates all of the effects of magnetic coupling between current loop 1 and any other current loop 2. We see from this expression that Z can be made arbitrarily small by reducing $\omega = 2\pi f$, the operating frequency. When Z is



$$Z = i\omega L_1 + \frac{\omega^2 M^2}{R_2 + i\omega L_2}$$

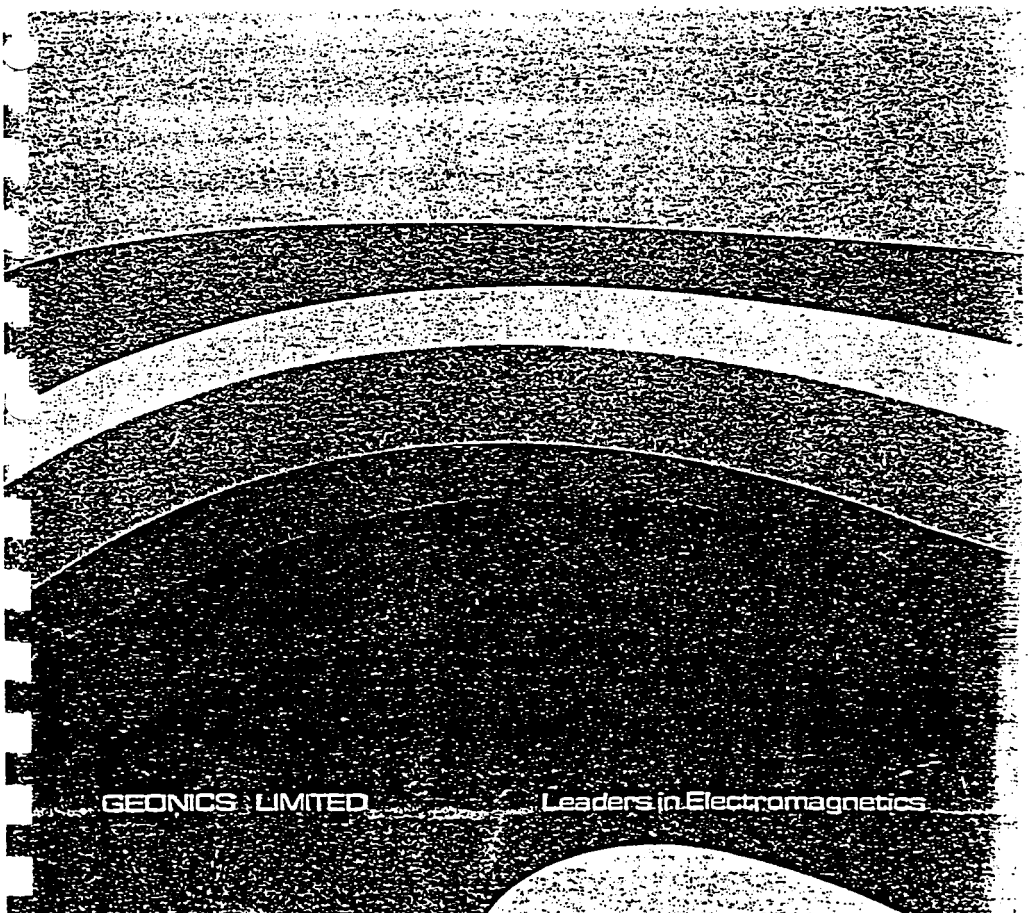
$$i_2 = \frac{e_s}{R_2 + Z}$$

FIGURE A3. Equivalent circuit for model of Figure A2.



Technical Note TN-5

ELECTRICAL CONDUCTIVITY OF SOILS AND ROCKS



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Technical Note TN-5

**ELECTRICAL CONDUCTIVITY
OF
SOILS AND ROCKS**

JD McNEILL

October, 1980

I. INTRODUCTION

It was near the beginning of this century that Conrad Schlumberger first employed the technique of mapping sub-surface geology by injecting electrical currents into the ground and mapping the resulting potential field distribution. Since that time measurement of terrain resistivity has been applied to a variety of geological problems. A partial list of applications includes the determination of rock lithology and bedrock depth; the location and mapping of aggregate and clay deposits; mapping groundwater extent and salinity; detecting pollution plumes in groundwater; mapping areas of high ice content in permafrost regions; locating geothermal areas; mapping archaeological sites, etc.

In many instances resistivity mapping provides definite geological information; however there are also cases where the results are uninterpretable since the "geological noise" is too high. A limitation of resistivity surveying is that the actual value of terrain resistivity itself is seldom diagnostic. As a result of this ambiguity we generally examine the variations of resistivity, either laterally or with depth, to outline the geological features of interest. But a problem arises in that conventional surveys are time-consuming to carry out and the area actually surveyed is often smaller than one might wish in order to fully ascertain the background against which the anomalous feature is to be defined. Furthermore, although conventional resistivity techniques sense to a characteristic depth (determined by the interelectrode spacing) resistivity inhomogeneities much smaller than that depth can, if they are located near the potential electrodes, yield large errors in the measurement and thus a noisy survey profile.

For these reasons application of resistivity surveys to engineering problems is not as common as it might be, particularly in North America. Such surveys have achieved success in Europe and are used more routinely.

It was an awareness both of the potential of resistivity measurements for solving geological problems and equally of some of the drawbacks of conventional resistivity mapping which lead Geonics Limited to develop two new lines of instrumentation employing electromagnetic techniques to measure terrain conductivity. In the first of these a sinusoidally varying magnetic field electromagnetically induces currents in the ground in such a manner that their amplitude is linearly proportional to the terrain conductivity (reciprocal of resistivity). The magnitude of these currents is determined by measuring the magnetic field which they in turn generate. Through the use of electromagnetic techniques, ground contact is avoided and with these patented instruments it is possible to map terrain conductivity virtually as fast as the operator(s) can walk; furthermore the sample volume is averaged in such a manner as to yield excellent resolution in conductivity. Two instruments have been developed by Geonics to cover the range of depths generally useful for engineering geophysics: (i) the EM31, one-man portable, has an effective penetration depth of 6 meters and (ii) the EM34-3, two-man portable, has stepwise selectable depths from 7.5 meters to 60 meters.

In the second approach the current flowing in a loop situated on the ground is abruptly terminated, inducing eddy currents in the ground which diffuse away from the transmitter loop in a manner controlled by the ground conductivity. In this case the dispersal of the currents is determined by measuring the transient decay of their magnetic field. Based on this principle, the Geonics EM37 can be used to determine the electrical properties of the earth to depths of several hundred meters.

These devices are assisting in the solution of many geological problems. With the renewed interest in resistivity it has become apparent that there is a requirement for a short note which discusses, from the point of view of survey interpretation, the various factors that influence terrain resistivity. For example, typical questions raised include (1) When are gravels more resistive than finer material? (2) What is the influence of the depth to water table? (3) It rained heavily last week; will this affect the measurements? (4) What are typical resistivities for the following soil types? etc., etc.

By describing the various factors that control the electrical conductivity/resistivity of soils and rocks under typical in-situ conditions this note will attempt to provide the technical background against which these questions can be answered and to thus give the operator greater confidence in his survey interpretation. The emphasis throughout this technical note is on those factors that influence the near-surface ground resistivity, particularly of soils.

The various topics are dealt with in the following sequence.

Section II: definition of conductivity & resistivity

Section III: description of relevant physical properties of soils and rocks

Section IV: relation of physical properties to the electrical conductivity

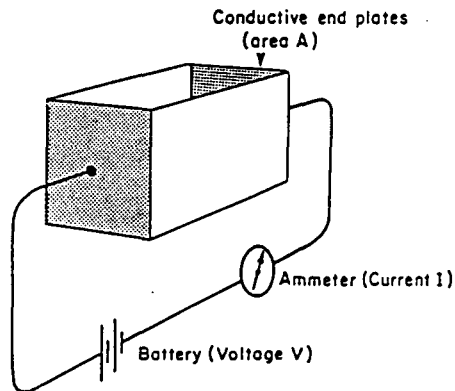
Section V: discussion of conventional resistivity measurements of soils in different climatic zones.

As mentioned above, this technical note is directed towards the user. Many readers will be familiar with the physical properties of soils and we ask their indulgence. None of the material on the electrical properties of soils is original; some has been taken verbatim from the references. However the compilation has been derived from many sources and we hope that it will prove useful.

II. RESISTIVITY/CONDUCTIVITY

Basically the electrical resistivity/conductivity of a substance is a measure of the difficulty/ease with which an electrical current can be made to flow through it. Suppose that we construct a tank with insulating sides and two conductive end plates as shown in Figure 1. The end plates are connected to a battery through an ammeter with which we measure the current flow through the sample. If the tank is empty there is of course no path by which the current can flow from one end plate to the other and the ammeter reads zero.

Suppose we fill the tank with a mixture of clean gravel and tap water. The ammeter will read a finite current and, depending on



RESISTIVITY (ρ)

$$\rho = \frac{RA}{L} \text{ ohm-metres}$$

$$\text{where } R = \frac{V}{I} \text{ ohms}$$

CONDUCTIVITY (σ)

$$\sigma = \frac{GL}{A} \text{ mhos/meter}$$

$$\text{where } G = \frac{I}{V} \text{ mhos}$$

FIGURE 1. Resistivity/conductivity tank.

various parameters to be discussed in Section IV, might indicate a current flow of a few thousandths of an ampere i.e. a few milliamperes, for a voltage of one volt. If the ammeter reads two milliamperes the electrical resistance of the material in the tank, given by the ratio of the voltage divided by the current, is 500 ohms.

If we now fill a number of different rectangular tanks, of different dimensions, with the same mixture we would find that the electrical resistance is proportional to the length of the tank and inversely proportional to the area of the conductive plate electrodes. The constant of proportionality, a property of the mixture only, independent of the tank dimensions, is defined as the electrical resistivity. In the MKS system it has the units of ohm-meters and is the electrical resistance measured on a cubic sample whose dimensions are all one meter. In the CGS system of units the resistivity is defined as the resistance across two opposite faces of a cubic sample one centimeter on each side and the units are ohm-centimeters. From the defining relation we see that, given the resistivity in ohm-centimeters, we must divide by 100 in order to get the resistivity in ohm-meters.

Suppose that we empty our tank (now assumed to be one meter on a side) and fill it with a mixture of clay saturated with water. We might find that the ammeter read a few tenths of an ampere, perhaps as much as half an ampere. The resistance would then be two ohms, the resistivity two ohm-meters. The range of resistivities displayed by unconsolidated materials at temperate ambient temperatures usually lies between one ohm-meter and one thousand ohm-meters; the resistivity of rocks can vary from a few tens of ohm-meters to as high as 100,000 ohm-meters, discussed in further detail in Section IV.

The reciprocal of the electrical resistivity of our sample is defined as the electrical conductivity. In the MKS system the unit of conductivity is the mho per meter and a resistivity of one ohm-meter exhibits a conductivity of one mho per meter, 100 ohm-meters is equivalent to a conductivity of 0.01 mhos per meter, etc. The electromagnetic instruments actually measure terrain conductivity rather than resistivity and for this reason much of the remainder of this technical note will be concerned with the conductivity of various terrain materials rather than the resistivity.

It was stated above that a resistivity of 100 ohm-meters corresponds to a conductivity of 0.01 mhos per meter. To avoid the inconvenience of having zeros immediately following the decimal point all conductivities will be expressed in millimhos per meter; a conductivity of .01 mhos per meter corresponds to 10 millimhos per meter, etc. This has the advantage that the range of resistivities from 1 to 1,000 ohm-meters is covered by the range of conductivities from 1,000 to 1 millimhos per meter and such numbers are easily handled. Table 1 lists the conversion from resistivity and conductivity in various units to conductivity in millimhos per meter.

In the experiments described above a direct current was employed. Had we used an alternating current and varied the frequency we might have discovered that the electrical properties of the sample varied with frequency. In reality soils and rocks are complex substances in which there are many (some poorly understood) mechanisms which govern the mode of current flow through the sample [1-5].

Fortunately, for materials with conductivity of the order of one to 1,000 millimhos per meter the electrical properties which control the current flow are relatively independent of frequency and the DC or low frequency conductivity measured with conventional resistivity equipment will be essentially the same as that measured using low frequency electromagnetic techniques.

Finally it should be noted that measurement of the electrical conductivity or resistivity of geological samples is, in reality, a very difficult procedure requiring much more complex equipment than the simple tank referred to above.

III. FACTORS AFFECTING TERRAIN CONDUCTIVITY

Most soil and rock minerals are electrical insulators of very high resistivity. However on rare occasions conductive minerals such as magnetite, specular hematite, carbon, graphite, pyrite and pyrrhotite occur in sufficient quantities in rocks to greatly increase their overall conductivity. This note assumes that such minerals are absent.

In general the conductivity is electrolytic and takes place through the moisture-filled pores and passages which are contained within the insulating matrix. The conductivity is therefore determined for both rocks and soils by

- (1) porosity: shape and size of pores, number, size and stage of interconnecting passages
- (2) the extent to which pores are filled by water i.e. the moisture content
- (3) concentration of dissolved electrolytes in the contained moisture
- (4) temperature and phase state of the porewater
- (5) amount and composition of colloids

Since the constituents, structure, and included electrolytes of most rock are of great importance in determining the conductivity, this section will discuss the various physical and chemical properties in sufficient detail to illustrate how they affect the material conductivity.

III. 1. Soil Constituents

Soils consist basically of four components namely: (i) mineral material (ii) organic material (iii) water and (iv) gases.

Figure 2 shows schematically the relative proportion of these components in an unsaturated loam soil (to be defined later). The illustration refers to a temperate zone soil; for humid region soils about 45% of the solid space may be occupied by clay minerals and 5% by organic matter.

The mineral fraction of a soil can be extremely variable, however some generalizations can be made based on grain size [6]:

sand size range - mostly quartz, small amounts of feldspar and mica present, all grains coated to some extent with iron and aluminum oxide (the rusty reddish-brown colour observed is due to the iron).

silt size range - quartz still dominant, less feldspar and mica than in the sand fraction, more iron and aluminum

TABLE 1. Resistivity/conductivity unit conversion factors.

mhos/meter $\times 1000$	→	millimhos/meter
$\frac{1000}{\text{ohm-meters}}$	→	millimhos/meter
$\frac{100,000}{\text{ohm-centimeters}}$	→	millimhos/meter
$\frac{\text{cgs electrostatic units}}{9 \times 10^9}$	→	millimhos/meter

Note: 1 Siemen (S) = 1 mho

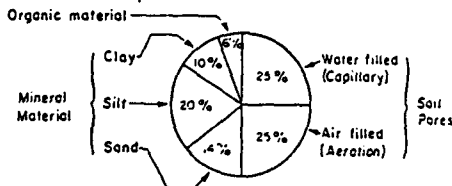


FIGURE 2. The volumetric composition of a loam soil when excess water has been removed. (On a weight basis the percentage composition of the dry soil would be: organic matter 4%, clay 22%, silt 44% and sand 30% (after J. K. Weber [6]).

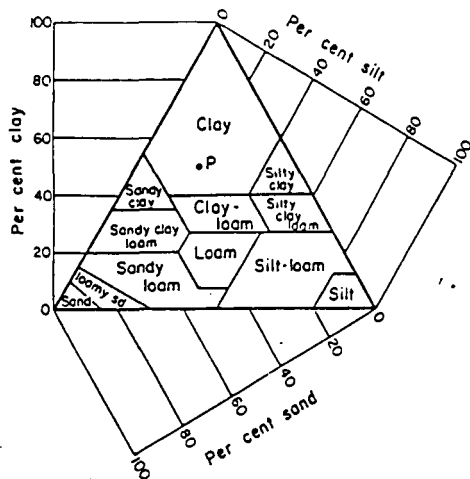


FIGURE 3. U.S. Department of Agriculture textural classification triangle with axes added. The point P represents a clay (soil) containing 50 per cent clay, 20 per cent silt, and 30 per cent sand (after D. Kirkham [7]).

oxides in the form of coatings due to the greater particle surface area on which the coatings may reside.

clay size range – finely divided quartz, feldspar, mica, iron and aluminum oxides, all of which appear in the coarser part of the clay fraction (0.002 millimeters – 0.001 millimeters in diameter); the finer part (less than 0.001 millimeters) is colloidal and consists mainly of layer silicates with smaller amounts of iron and aluminum oxides.

As indicated above soils are classified on the basis of texture or grain size independently of the mineralogical content of each particle size component. Sand is defined as particles with diameters between 0.05 millimeters and 2 millimeters, silt has diameters between 0.002 millimeters and 0.05 millimeters and clay has diameters less than 0.002 millimeters. Figure 3 shows the classification triangle for various types of soil and is useful as it allows an estimate of the clay content which often consists essentially of clay minerals which affect the soil conductivity.

The minerals in the sand and silt fractions of the soil are electrically neutral and are generally excellent insulators. Completely dry clay is also an insulator but the introduction of moisture changes the situation radically. Clay consists of microscopically fine particles

TABLE 3. The cation exchange capacity (CEC) and colloid content of five soils of different textures (after L. R. Webber [6]).

Soil Texture	Organic Matter %	Clay %	CEC* me/100 g
Sand	1.7	7	6.3
Sandy loam	3.2	13.2	13.7
Loam	4.9	16.8	20.2
Silt loam	5.4	18.4	24.0
Clay loam	5.5	31.2	27.2

*The cation exchange capacity of a soil is expressed in terms of milliequivalents per 100 grams of soil (meq/100 g). A milliequivalent is defined as 1.0 milligram of hydrogen or the amount of any other element that will combine with or displace it. The milliequivalent weight of any element may be found as follows:

$$\frac{\text{atomic weight of element}}{\text{valence of element} \times 1000} = \text{milliequivalent weight}$$

exhibiting a sheet-like structure, for which reason clays are often called "layer silicates". Composed of stable secondary minerals that have formed as a result of weathering of primary minerals such as feldspar, mica, etc. the particles are so fine-grained that they are described as micro-crystals.

Their crystalline structure is such that, as a result of crystal imperfections, the surface appears to be negatively charged [5]. During the formation of the clay through weathering, positive charges (cations) are adsorbed to the surface. These cations (typically Ca, Mg, H, K, Na, NH₄) are loosely held to the surface and can subsequently be exchanged for other cations or essentially go into solution should the clay be mixed with water. For this reason they are called exchangeable ions and the cation exchange capacity (CEC) of the soil is a measure of the number of cations that are required to neutralize the clay particle as a whole i.e. the weight of ions in milliequivalents adsorbed per 100 grams of clay.

The exchange capacities of some common clays are given in Table 2 and of some different soil textures in Table 3. It is seen from Table 3 that the cation exchange capacity increases with clay content and from Table 2 that this will depend on the type of clay.

Clay minerals are not the only materials which have cation exchange capacity; indeed any fine-grained mineral including quartz displays this property. The special significance of clay in this respect is that, because of the extremely small particle size, the surface area per unit volume of clay is very large and a great many ions are adsorbed. It will be seen in the next section that these adsorbed ions can contribute appreciably to the soil conductivity which thus becomes a function of the clay content.

The organic matter includes the remains of plant and animal life in the soil. The end products of decay accumulate as a blackish, finely-divided colloidal substance known as humus [6] which has large surface area per unit volume, takes up large amounts of water and can develop a negative electrical charge of varying intensity. Little is known of the effects of such colloidal characteristics on the electrical conductivity of humus but they may be significant.

The important influence of soil moisture on the electrical conductivity of soils is discussed at length in a later section.

The direct effect of soil gases on conductivity is negligible, however the indirect effect of CO₂ is important and is also described in a later section.

III. 2. Formation of Soils [8] and Soil Profiles [9]

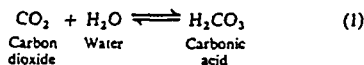
Soils are the result of mechanical, chemical, and biological weathering processes acting on surficial materials in such a way as to grossly alter their physical and chemical properties. In the process of decomposition of parent materials new and stable substances such as clay minerals and humic materials are formed.

As an example of the weathering process consider the chemical

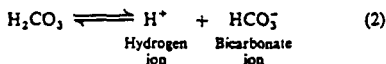
TABLE 2. Exchange capacity of common clays (after Keller and Frischknecht [5]).

Clay	Exchange Capacity
Kaolinite	3 to 15 m-equiv/100 g
Halloysite . 2H ₂ O	5 to 10
Halloysite . 4H ₂ O	40 to 50
Montmorillonite	80 to 150
Illite	10 to 40
Vermiculite	100 to 150
Chlorite	10 to 40
Attapulgite	20 to 30

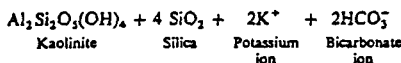
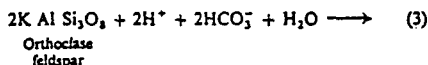
athering of orthoclase feldspar (an important constituent of granites and other intrusive rocks) to kaolinite, a clay. Atmospheric carbon dioxide is slightly soluble in water so as to form carbonic acid.



A small amount of the carbonic acid dissociates so as to produce a hydrogen ion and a bicarbonate ion



The bicarbonate ion and water combine with the feldspar to form kaolinite, dissolved silica, free potassium ions and free bicarbonate ions



The formula for kaolinite can also be written in the following manner



to show that it consists of alumina, silica, and water.

These equations illustrate three important points: (i) The potassium and silica produced by dissolving the feldspar appear as dissolved material. Note that the potassium, converted to a potassium ion, goes into solution and may or may not be present depending on the drainage. (ii) Water is used up in the reaction; it is absorbed into the kaolinite structure. (iii) Hydrogen ions are used up in the reaction and the solution becomes more basic (i.e. less acidic) as the reaction proceeds. All of the reactions described herein affect the electrical properties of the soil by varying either the clay content or the ionic type and concentration in the soil water.

Now let us examine how various factors control this weathering process. The production of soil is a positive feedback process in that once a thin veneer of soil forms, the parent material weathers more rapidly, and more soil is formed. Suppose for example that we have a thin layer of soil on granite. This layer retains moisture which supplies both water that converts carbon dioxide to carbonic acid and water that is hydrated to form kaolinite. Other acids present in the soil contribute additional supplies of hydrogen ion to convert more feldspar to kaolinite as the equations indicate. Plant roots and the process of bacterial decay produce quantities of carbon dioxide to yield more carbonic acid (the amount of carbon dioxide in soil can be as much as ten times greater than that of rainwater, which makes soil water a particularly efficient dissolver of feldspars).

The speed of weathering is a function of climatic type since the rate at which chemical reactions proceed increases with temperature and, more importantly, biological process also proceed faster with higher temperature. Furthermore, since water is needed for the weathering reactions and vegetation grows more lushly in humid climates weathering is most intense in tropical climates which are wet and warm. The surface water resulting from rainfall percolates downwards through the soil, which is more or less permeable, and as it passes reacts chemically with minerals within the soil. The higher the rainfall the more water is available and the more the

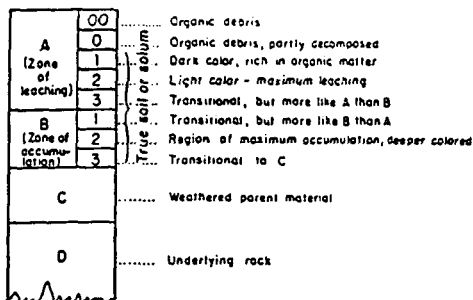


FIGURE 4. Normal or zonal soil profile (after G.B. Maxey [9]).

minerals will react with the undersaturated water. The longer the time the water percolates through, the more time it has to react chemically and again the higher the temperature, the faster the reaction rates.

As a result of these factors tropical soils tend to be thick. Well drained soils tend to be devoid of most unstable minerals. Arid soils contain a variety of minerals. The clay minerals which form are a function of temperature and humidity and there is a tendency for different clays to form in the various climatic belts [10].

As long as there is rainfall and the temperatures are not too low there is a tendency for soils to form a layered structure. In general three different regions or horizons are recognized as we move down through the soil profile, usually designated by the letters A, B, and C. These horizons can vary in thickness from a centimeter to several meters or tens of meters and differ in colour, texture, structure, and other properties.

The chief characteristics of a profile are illustrated in Figure 4. The A horizon, closest to the surface, is the most intensely weathered and has the soluble minerals leached out and most other minerals altered. This layer usually contains much humus, which contributes to its dark colour. Structurally it is friable, granular, or platy. When either friable or granular this horizon is permeable, much more so than the underlying B horizon which is generally a zone of clay accumulation, some of which was formed in-situ and some of which was transported downwards from the A zone by the soil water. The B horizon generally displays a vertical structure in widely or closely spaced joints and it is this horizon that exerts the greatest influence on water movement vertically downwards. When the clay is dry these joints allow rapid downwards movement but when the clay is wet it expands and can close the joints to make the layer impermeable, which may in turn cause the A horizon to become saturated for appreciable periods of time. The C horizon consists of less-weathered parent material and is usually relatively permeable [9].

This layering, with its relatively clay-free A horizon and clay-rich B horizon, greatly influences the vertical profile of electrical conductivity which will be seen to be a function of both clay and water content.

III. 3. Soil Moisture

It was noted at the beginning of this section that the electrical conductivity of soils and rocks was primarily electrolytic and took place through the moisture filled pores and passages which lie within the matrix of insulating minerals. For this reason a knowledge of the way in which soil moisture is distributed in typical terrain is important in understanding terrain conductivity.

When rainwater or irrigation falls on the surface of the soil a fraction runs off directly as surface runoff and the remainder percolates directly into the soil. A fraction of this moisture is retained by the soil, the remainder moves vertically downwards under the force

of gravity until it reaches the water table. In a soil moisture profile it is generally possible to distinguish four stages of moisture occurrence depending on the relative continuity of the moisture films across the soil grains [11] as shown in Figure 5. In the uppermost region we have the pendular state in which the pore space is largely filled with water vapour. The actual liquid water exists only in very small isolated rings around the grain contacts and a continuous path does not exist between the various moisture occurrences. At greater depth we have the funicular stage in which the pendular rings have coalesced to the point where the liquid films have just become continuous throughout the pore space and entirely enclose or encapsulate the vapour phase. Across the sample the moisture path is now continuous. Again further down, in the capillary stage, all pore spaces are occupied by liquid but the liquid pressure within the pores is less than the total pressure caused by gravity since capillary action within the fine pore spaces has caused the moisture to ascend into these pore spaces. Capillary rise, determined effectively by pore size and type, seldom exceeds several meters. Finally we arrive at the phreatic surface (water table) at which the atmospheric pressure is in equilibrium with the hydrostatic pressure. All pores within the phreatic surface are completely filled with liquid under hydrostatic pressure and this is the region of groundwater, also known as the zone of saturation. The three regions above the phreatic surface are collectively referred to as soil moisture, suspended water, vadose water, or zone of aeration.

Figure 5 also illustrates typically the fraction of pore space filled by liquid in the different zones.

A term that is often used in discussing the moisture content of soils is field capacity. Suppose that the water table is very deep below a surface soil of homogenous nature. After a heavy rainfall or irrigation the moisture content reaches a quasi-equilibrium condition in one to three days. As the water percolates down through the soil on its way to the water table capillary forces will retain a certain fraction of the moisture in the small pore spaces. This is the field

TABLE 4. Permeability of soils (after D. K. Todd [12])

Soil class	Coarse gravel	Coarse sand, 40% to 60% of coarse grains 10 grains	Fine sand, 60% to 80% of coarse grains 10 grains	Silt and clay, 80% to 100%	Unconsolidated silts
Flow characteristics	Coarse channels				
Permeability (cm/sec)	10 ⁻¹	10 ⁻²	10 ⁻³	10 ⁻⁴	10 ⁻⁵

capacity and it is the moisture content of the soil after the gravitational water has been removed by deep seepage.

Note that in the event that we have a layered situation in which a soil with very fine pores overlies more granular material, both being far removed from the water table, moisture content of the upper layer will be many times that of the lower which, as will be seen later, affects the electrical conductivity.

Groundwater is free to move laterally with velocities ranging from a meter or more per day to less than a meter per year, depending on the hydraulic pressure differential and the permeability of the material. Table 4 illustrates the permeability of various materials in gallons per day per square foot of cross-section per foot of hydraulic head per lateral foot of distance. It is seen from the Table that for soil materials a factor of 10⁵ separates permeable from impermeable materials. The saturated hydraulic conductivity decreases with clay content, increasing compaction, and decreasing radius of the soil pores.

The laws of hydraulic movement of water produce a water table which in general is not horizontal and in fact is often a subdued version of the local topography, all other factors being equal. Two examples are shown in Figure 6; for the humid zone the moisture moves from topographic high regions down to the draining streams whereas in an arid zone the moisture moves downwards away from the streams.

The wide ranges of permeability greatly influence the final profile of the water table or phreatic zone sometimes leading, for example, to the occurrence of a perched water table as shown in Figure 7. Again such layering will be seen subsequently to influence the electrical properties.

It will be shown in Section IV that the electrical conductivity of soils and rocks depends on the porosity and on the degree to which the pores are filled with moisture. Figure 2 showed the volumetric composition of a loam soil from which the excess water has been removed i.e. the soil is undersaturated. We see that in this temperate zone example approximately 50% of the soil is occupied by either gas or capillary water. This division into about 50% solids is fairly typical of most soils.

If all of the moisture in a soil sample is removed by drying, the ratio of the empty volume to the total volume of the soil matrix is known as the soil porosity, a parameter that is relevant to the soil conductivity. Table 5 illustrates the porosity for a variety of typical terrain materials.

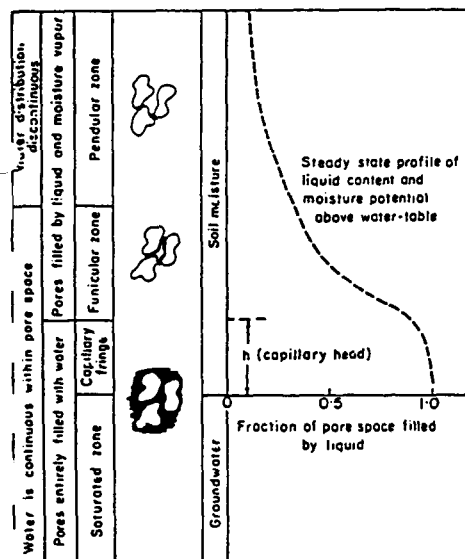


FIGURE 5. Liquid occurrence in soils (after P. Meyboom [11]).

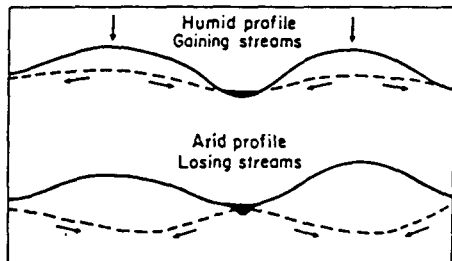


FIGURE 6. Profiles of water tables in arid and humid zones (after G. B. Muxey [9]).

Then the number of gram equivalent weights of sodium per 10^6 cm^3 water is given by

$$C_{Na} = \frac{\text{weight of Na per } 10^6 \text{ cm}^3 \text{ H}_2\text{O}}{\text{gram equivalent weight}} = \frac{23/58}{23} = \frac{1}{58}$$

and that for chlorine is given by

$$C_{Cl} = \frac{\text{weight of Cl per } 10^6 \text{ cm}^3 \text{ H}_2\text{O}}{\text{gram equivalent weight}} = \frac{35/58}{35} = \frac{1}{58}$$

Using the data from Table 7 for the mobilities at 25°C we find that the conductivity for one gram of sodium chloride in 10^6 cm^3 of water is given by

$$\sigma = 96500 \left[\frac{1}{58} \times 5.2 \times 10^8 + \frac{1}{58} \times 7.9 \times 10^8 \right] = 0.00022 \text{ mho/m. (5)}$$

The addition of only one part per million of sodium chloride by eight has produced the appreciable conductivity of 0.22 millimhos per meter.

The concentration of dissolved salts in natural groundwaters is substantially higher than one part per million, as a result of which their conductivity is much greater than 0.22 millimhos per meter. For example Table 8 illustrates the contribution of various ions to the measured conductivity of three of the Great Lakes. Lakes Erie and Huron which both occur in regions of Paleozoic carbonate rocks contain more dissolved salts than Lake Superior which is situated largely in Precambrian crystalline rocks, and this is reflected in the higher conductivities of the first two lakes. The bottom line of the table gives the measured conductivity; agreement with the calculated values using the above equations is good.

The following data from Heiland [14] illustrates typical values for the conductivity of various natural waters.

- Meteoric waters, derived from precipitation; 1 to 30 millimhos per meter.
- Surface waters (lakes, rivers) vary from 0.3 millimhos per meter for very pure water to as large as 10,000 millimhos per meter for salt lakes: Surface waters in districts of igneous rocks are estimated to range from 2 to 30 millimhos per meter; surface waters in areas of sedimentary rocks vary from 10 to 100 millimhos per meter (compare the Great Lakes above).
- Soil waters (discharged into the atmosphere by evaporation) may be as large as 10,000 millimhos per meter but their average is around 10 millimhos per meter.
- Normal groundwater in areas of igneous rock ranges from 6 to 30

millimhos per meter and in areas of sedimentary rocks to as large as 1,000 millimhos per meter.

- Mine waters (copper, zinc, etc., sulfates) are of high conductivity, generally not less than 3,000 millimhos per meter.

The temperature dependence of the electrical conductivity of the electrolyte is almost entirely due to the temperature dependence of the viscosity of the liquid, which in turn directly affects the ionic mobility. The variation of either quantity with temperature is approximately linear over normal ambient temperatures. The temperature coefficient for a sodium chloride solution is 0.022 which value applies approximately to most other ions so that the electrolyte conductivity for a temperature other than 25°C is given by

$$\sigma(T) = \sigma(25^\circ\text{C})[1 + \beta(T - 25^\circ)]$$

where $\beta = 2.2 \times 10^{-2} \text{ per } ^\circ\text{C}$

T = temperature ($^\circ\text{C}$) at which conductivity is to be calculated.

A change of conductivity of 2.2% per degree centigrade implies that a change in temperature of 40°C will cause the conductivity to nearly double. The effect of temperature is illustrated in Figure 8 which shows calculated conductivity of four of the Great Lakes as a function of season.

The change of conductivity with temperature and therefore with season is not negligible and this applies equally well to ground conductivity over the normal range of ambient temperatures.

IV. 2. Conductivity of Saturated Clean (Clay-Free) Mixtures

We have examined how the electrical conductivity of the electrolyte in our tank varies with the concentration of dissolved salts and with temperature. Let us now start to fill the tank with perfectly insulating spheres of uniform radius (for example uniform pebbles). Assume for the time being that we can maintain these spheres uniformly dispersed throughout the solution. Since the spheres are insulating, the electrical current will find it more difficult to cross the tank and the conductivity will be reduced. A relationship was derived by Maxwell for the conductivity σ_m of a mixture consisting of a medium with conductivity σ_i in which spherical grains of conduc-

TABLE 8. Conductivity of Great Lakes with contribution of the various ions (after L. H. Doherty [15])

Ion	Lake Erie	Lake Huron	Lake Superior
HCO_3	5.8	4.1	2.6
Ca	10.1	7.3	3.8
Mg	3.6	3.2	2.4
Na	1.8	0.6	0.2
Cl	3.9	1.2	0.2
SO_4	3.4	1.8	0.3
Calculated Conductivity (mmhos/m)	28.6	18.2	9.5
Measured Conductivity (mmhos/m)	26.7	18.2	8.4

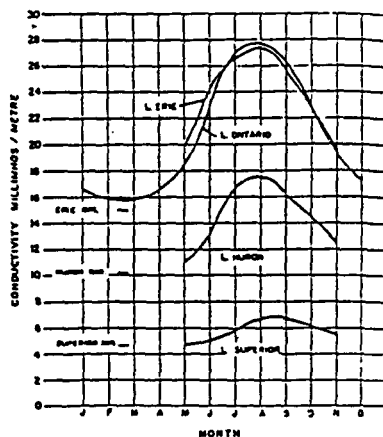


FIGURE 8. Seasonal variation of conductivity of Lakes Ontario, Erie, Huron and Superior (after L. H. Doherty [15]).

tivity σ_1 (here assumed to be zero) are imbedded in regular arrangement and in such a manner that their spacing is large compared with their radius [14]. Letting n be the porosity of the mixture (defined as the ratio of the volume of solution only, divided by the total volume of the solution including the spheres) Maxwell showed that the conductivity of the mixture was given by

$$\frac{\sigma_2}{\sigma_1} = \frac{2n}{3-n} \quad (7)$$

In Section II it was shown that the conductance of a current path is directly proportional to the cross-sectional area and inversely proportional to the length. The addition of insulating spheres to the tank tends to reduce the cross-sectional area available for current flow and to increase the effective length of the current paths, reducing the overall conductivity.

This dilute mixture does not resemble a soil so we continue to add insulating spheres until the tank is full. The condition for Maxwell's derivation, that the inter-particle distance be large compared with the particle radius, is invalid and the above equation can no longer be expected to apply. It turns out however that an almost equally simple empirical relationship called Archie's Law [5] is applicable to clean (i.e. clay-free) saturated mixtures. This equation is

$$\frac{\sigma_2}{\sigma_1} = n^m \quad (8)$$

where n is the fractional porosity defined above and m is a constant. The reciprocal quantity p_2/p_1 is often called the formation factor (FF) of the rock or soil sample.

Originally derived from resistivity measurements on samples of consolidated porous rock, this equation also applies to a variety of unconsolidated materials. That such a simple law should give excellent results for a variety of both consolidated and unconsolidated materials with widely differing porosities is a surprise and indeed the underlying reasons for its success are not well understood [4]. It has been established [16] that in the case of marine sands the exponent m is dependent on the shape of the particles, increasing as they become less spherical (i.e. more platy) and that variations in the size of the particles and in the dispersion of sizes appear to have a very small effect.

Values of m are listed in Table 9 where it is seen that m varies from 1.2 for insulating spheres to 1.85 for very platy fragments of shell. Samples of natural sands have values in the range 1.4 to 1.6. The value of 1.85 for shell fragments is in good agreement with earlier measurements made on kaolinite particles and marine illite clays. This fact may or may not be significant for clays in normal soils depending on the extent to which ion exchange effects also contribute to the conductivity as discussed further on in this section.

Archie's Law for various exponents is plotted in Figure 9 along with Maxwell's Law. Over the range of porosities of most unconsolidated terrain materials (20% to 70% - see Tables 5 and 6) the different exponents do not greatly affect the mixture conductivity. Interestingly enough, Maxwell's Law gives excellent agreement with Archie's Law with exponent $m = 1.3$ over all ranges of porosity.

It would appear that for relatively clay-free substances located beneath the water table (so that the mixture is completely saturated) the primary matrix property measured through the electrical conductivity is the porosity of the matrix, essentially independent of the particle size or the particle size distribution. This explains why it is a relatively difficult matter using resistivity techniques to distinguish between sand and gravel. As long as the porosity is the same for both sand and gravel the resistivity or conductivity contrast may be quite small.

Another point is that if the porosity is assumed to vary from 20% to 70% the conductivity of the mixture varies by a factor of approximately 8, depending somewhat on which value of exponent is

TABLE 9. The effect of particle shape on the FF/n relation using artificial samples of decreasing sphericity. (after P. D. Jackson et al. [16])

Sample no.	Mean size ϕ^*	Spread of sizes	Sphericity	Best fit Archie line $FF = n^{-m}$ $m \pm 0.01$
Spheres	0.38	0.17	1.0	1.20
Rounded sand	0.38	0.17	0.83	1.40
Shaley sand	0.50	0.34	0.78	1.52
Shell fragments	0.38	0.17	0.5	1.85

* $\phi = -\log_2$ (diameter in mm).

adopted. Even in clean mixtures we do not expect a large range of conductivities and experimentally this has been confirmed.

Figure 9 is useful for the following type of calculation: in many glacial deposits gravel appears as pebbles (of the order of a few centimeters in diameter) dispersed throughout a mixture of finer relatively clean sand. How does the presence of this gravel modify the mixture conductivity? To obtain an approximate answer let us assume that the sand itself has a porosity of 30%. Using Archie's Law with $m = 1.6$ the electrical conductivity of the sand/water mixture will be 15% of the conductivity of the water. Assuming a water conductivity of 20 millimhos per meter gives a conductivity for the mixture of 3 millimhos per meter, a not uncommon value. We use this value in conjunction with Figure 9 to determine the effect of introducing pebbles into the mixture. If for example the pebbles occupy 50% of the volume the conductivity of the mixture will fall to one-third or approximately 1 millimho per meter, also a value that commonly occurs in actual practice. As will be seen later the addition of a relatively small amount of clay can increase these numbers by virtue of increasing the conductivity of the pore water but the relative values may well be similar.

IV. 3. Conductivity of Unsaturated Clean Mixtures

For Archie's Law to apply the material must be fully saturated with fluid. If the mixture is partially saturated the conductivities will be decreased since gas or air bubbles act as insulating particles to further impede the current flow. In the funicular stage of soil moisture the pendular rings have coalesced so that the liquid films are continuous throughout the pore space but only a fraction of the available pore space is filled with water. This moisture varies with time and temperature as a result of drainage, evaporation, and loss of water to plant roots.

In the event that the soil is partially desaturated the following approximate expression applies [5]

$$\frac{\sigma_2}{\sigma_1} = s^n \quad (9)$$

where s is the fraction of total pore volume filled with electrolyte and n is a parameter experimentally determined to be approximately 2. This expression is equivalent to Archie's Law with $m = 2$ and indicates as seen from Figure 9 that if a small fraction of the total pore volume is filled with water the conductivity can be very low.

More recent work on soils has shown that the electrical conductivity varies as follows with moisture content when the soil is partially desaturated [17]

$$\sigma_2 = \sigma_1 \theta (a\theta + b) + \sigma_1 \quad (10)$$

where θ is the volumetric water content (cm^3 of water per cm^3 of soil), a and b are constants which depend on the soil texture, and σ_1 is a contribution from "surface conductivity" which will be discussed under Colloidal Conductivity. The values of the empirically determined constants a and b for a variety of soils are shown in

TABLE 10. Measured moisture content constants and surface conductivities (after J. D. Rhoades et al. [17])

Soil type	a	b	σ_s
Pachappa fsl	1.382	-0.093	0.18
Indio vfst	1.287	-0.116	0.25
Waukena l	1.403	-0.064	0.40
Domino cl	2.134	-0.245	0.40

increasing depth the conductivity rises rapidly as the moisture films become continuous, then more slowly as the available pore volume starts to fill, eventually saturating with complete filling of the pores. This situation will be rendered even more complex if the porosity itself is, as is often the case, a function of depth as suggested by the profile of Figure 4.

IV. 5. Colloidal Conductivity

It will be recalled that for clays the cation exchange capacity (CEC) was a measure of the number of ions adsorbed to the surface of clay particles. When the clay particles are immersed in a liquid there is evidence to show that these adsorbed ions can partially dissociate themselves from the clay particles and become available for ionic conductivity. Since the ion exchange capacity of clays can be great due to their large surface area many ions may be supplied for electrical conductivity; the addition of a small amount of clay to an otherwise clean mixture can substantially increase the electrical conductivity.

The addition of clay appears to affect the electrical conductivity of the mixture in two ways. Repeating equation (10)

$$\sigma_s = \sigma_1 \theta(a\theta + b) + \sigma_s \quad (10)$$

the added ions increase the value of σ_1 above the value that the porewater would have in the absence of the clay. Furthermore although dry clay is highly resistive, as soon as a thin layer of moisture (perhaps only a few molecules thick) surrounds the clay particles ion movement across the surface of the clay particle within the cloud of adsorbed ions may occur. This surface contribution to the conductivity, essentially independent of the moisture content, is the second term in the equation. It will be most significant at low moisture contents.

The contribution to σ_1 from clay content will be most evident for soils in which the porewater is relatively pure (and therefore has low conductivity) and will be least effective in soils having highly saline porewater. For either contribution we should expect that clays with higher CEC will produce more conductive soils and a comparison of Table 11 with Table 2 shows that this is the case.

In summary, in areas where the soil porewater is not particularly saline the electrical properties of the soil may be strongly influenced by and indeed in some cases completely dominated by the amount and type of clay minerals present. The possible influence of clay materials should always be kept in mind. It is also possible that a similar effect arises as a result of the colloidal properties of humus which might be important in tropical climates where the humus layer is well leached and contains few clay minerals.

IV. 6. Effects of Freezing on Soil Conductivity

Suppose that we take our tank, filled with a clean sand/water mixture, and start to lower the temperature. The electrical conductivity of the mixture will decrease in exactly the same way that the conductivity of the electrolyte does. When the temperature reaches 0°C the water freezes and since the conductivity of ice is extremely low the conductivity of the mixture falls essentially to zero.

Now suppose that in addition to the sand some clay is introduced to the mixture and the temperature is again reduced. As the temperature passes through 0°C some of the water freezes; however the

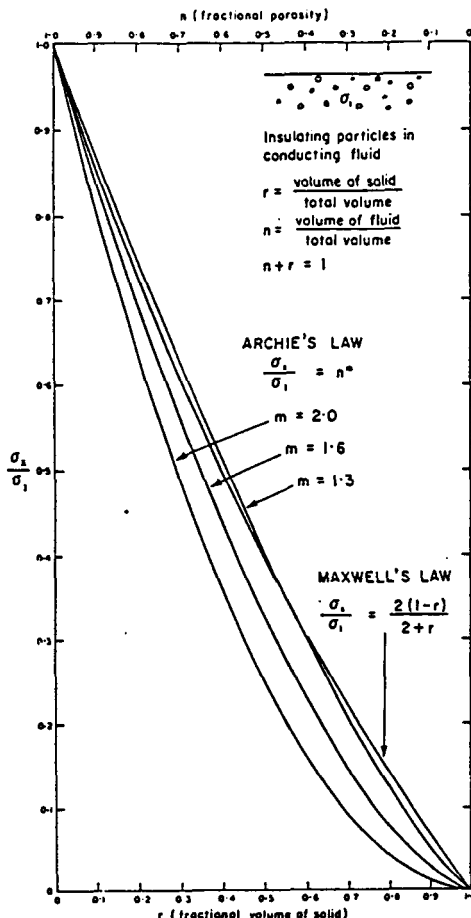


FIGURE 9. Graph of Archie's Law and Maxwell's Law.

Table 10 from which it is seen that except for very low moisture the relationship is essentially a square law with moisture content.

It has been suggested by Rhoades et al [17] that it might be possible to estimate the values of σ_s , a, and b on the basis of soil texture and mineralogy and, given a measurement of the bulk soil conductivity and the moisture content, to determine the conductivity of the fluid and thus salinity.

IV. 4. Electrical Layering Arising from Soil Moisture

From the preceding paragraphs we observe that, even if the soil material is physically homogeneous, for example consists of clean well-sorted silt, as soon as we introduce soil moisture of finite electrical conductivity we have established a medium in which the electrical conductivity can vary strongly with depth. Near surface where the moisture content is low the conductivity is also low. With

TABLE 11. Physical properties of typical Upland and Lowland soils in western Puerto Rico (after J. W. Walker et al. [18])

Topographic location	Sample no.	Moisture (%)	Conductivity (mmho/m)	Clastic material (%)	Clay fraction (%)	Clay type(s) (%)
Uplands	1	42	16.0	21	79	100% kaolinite
Uplands	2	46	1.2	42	58	100% kaolinite
Uplands	3	25	1.0	5	95	100% kaolinite
Lowlands	4	21	65.0	66	34	100% montmorillonite
Lowlands	5	32	169.0	39	61	60% montmorillonite 40% kaolinite
Lowlands	6	25	269.0	15	85	90% montmorillonite 10% kaolinite

electrical field of the adsorbed ions on the clay particles locally orients the nearby water molecules to prevent their freezing [17]. Furthermore as the solvent freezes there is a tendency for the impurity ions to stay within the liquid fraction and the actual electrical conductivity of the remaining liquid water increases with decreasing temperature. The net result is that mixtures containing clay or silts tend to have an electrical conductivity which decreases relatively slowly with temperature as the temperature passes through 0°C and indeed to retain a moderate conductivity even at temperatures well below freezing as illustrated in Figure 10.

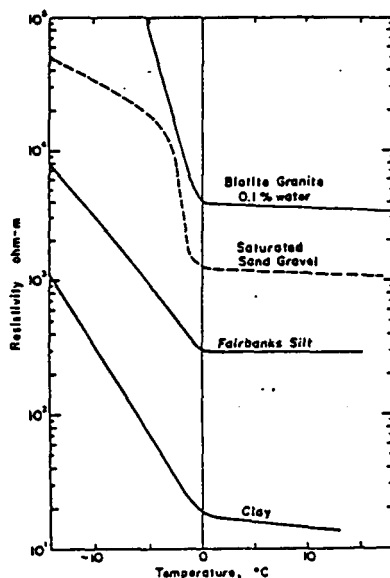


FIGURE 10. Resistivities for several soils and one rock type as a function of temperature (after Hoeksma and McNeill [20]).

IV. 7. Electrical Properties of Rocks

As with unconsolidated materials the matrix of most water-bearing rocks is insulating and the electrical conductivity is also electrolytic. A major difference between unconsolidated sediments and rocks lies in the types of pore geometries. For example in sedimentary rocks the porosity is generally inter-granular and consists of voids still remaining from the compaction process. In igneous rocks the porosity of the rock itself may be extremely small, however moisture circulates through fractures in the rock which are the result of mechanical breakage. These cracks are called joints and may be sufficiently large and/or numerous that their presence completely dominates the electrical conductivity. Such jointing may also play a major role in the conductivity of the more impermeable sedimentary rocks. When large joints are present the local conductivity may be expected to vary strongly with position. A third form of porosity which may not make a major contribution to electrical conductivity as normally measured is vugular porosity. It consists of cavities (as might be caused by solutions circulating in limestone) interconnected by small pores which dominate the D.C. electrical behavior. It should be noted that in the case of electromagnetic excitation of currents direct electrical (ohmic) inter-cavity connection is not necessary and appreciable response to this excitation may come from the fluid in each cavity separately.

Table 12 illustrates normal porosity for various rock types. It is seen that the total porosity can be very small, of the order of a few percent, and we would therefore expect, particularly for unfractured Precambrian igneous rocks and high-rank metamorphosed rocks, the electrical conductivity to be very low, as is generally the case. On the other hand shales may be relatively porous and in addition the conductivity of the pore water may be high as a result of ion exchange effects. For this reason shales can be as conductive as 30 to 40 millimhos per meter and in some cases even higher.

Since in general the porosities are smaller for consolidated materials and furthermore at this end of the scale the various exponents in Archie's Law have a large effect, it is difficult to predict the electrical properties of any given rock type.

There is another feature which occurs in sedimentary rocks and which is of importance in determining their electrical characteristics. Being depositional in nature these rocks are layered and the electrical conductivity perpendicular to the bedding planes may be less than the conductivity parallel to the planes.

This feature is illustrated in Table 13 which lists the coefficient of anisotropy for various layered rocks. The coefficient is defined in Figure 11.

Another type of rock conductor called a structural conductor [21] occurs as a result of fracturing and is often linear in shape. Such conductors arise in the interior of faults, shear zones, contact frac-

TABLE 12. Normal ranges in porosity for rocks (after Keller and Frischknecht [5])

Rock type	Intergranular porosity (%)	Joint porosity (%)	Vugular porosity *
Paleozoic sandstones and shale	5-30	0-1	0
Paleozoic limestones	2-10	0-2	0
Paleozoic clastic volcanics	5-30	0-2	0
Post-Paleozoic sandstones and shale	10-40	0	0
Post-Paleozoic limestones	4-20	0-2	0
Post-Paleozoic clastic volcanics	10-60	0	0
Precambrian sediments and low-rank metamorphosed sediments	1-8	0-2	0
Precambrian igneous rocks and high-rank metamorphic rocks	0-2	0-2	0
Slate recent igneous rocks	0-10	0-2	0

*Vugular porosity accounts for an appreciable total porosity only in rare cases.

TABLE 13. Coefficients of anisotropy for layered rock (after Keller and Frischknecht [5])

Rock type	Coefficient of anisotropy
Volcanic tuff, Eocene and younger, from Nevada	1.10-1.20
Alluvium, thick sections from the southwestern United States	1.02-1.10
Interbedded limestones and limey shales from northeastern Colorado	2.0-3.0
Interbedded anhydrite and shale, northeastern Colorado	4.0-7.5
Massive shale beds	1.01-1.05
Interbedded shale and sandstone	1.05-1.15
Baked shale or low-rank slate	1.10-1.60
Slates	1.40-2.25
Bituminous coal and mudstone	1.7-2.6
Anthracite coal and associated rocks	2.0-2.6
Graphitic slate	2.0-2.8

ture zones, etc., where the rock material has been ground into small particles which allows increased circulation of groundwater resulting in enhanced weathering to produce clay minerals. Structural conductors occur in a wide range of sizes.

V. EXAMPLES OF CONVENTIONAL MEASUREMENTS OF SOIL AND ROCK RESISTIVITIES

The material in this section gives a broad indication as to the range of resistivity or conductivity which might be encountered in various terrain materials in various climatic zones. Extreme caution must be exercised in employing these values for anything other than a rough guide as to anticipated survey results.

As an introduction Table 14 illustrates the resistivities of igneous and metamorphic rocks as given by Telford et al [22]. Table 15 lists resistivities of sediments from the same source. Table 6, from Heiland [14], lists the ratio ρ_x/ρ_z as defined in Section IV for various consolidated and unconsolidated materials.

V.1. Temperate Zones

Table 16 records the ranges of resistivity compiled for different terrain materials from a variety of survey and laboratory measurements, from Culley et al [23].

Table 17 from Sellman et al [24] shows survey data over different soil types made with three different measurement techniques (i) radio-frequency magneto-telluric at approximately 300 kHz, (ii)

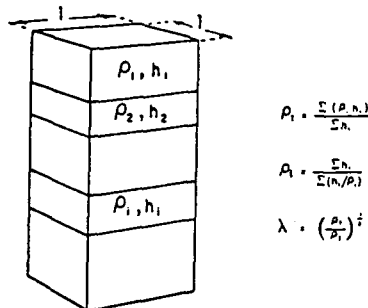


FIGURE 11. Definition of coefficient of anisotropy (λ) for layered sediments (after Keller and Frischknecht [5]).

low-frequency magnetically induced currents using dipole transmitter and receiver at 10 kHz, and (iii) standard Wenner array at DC. The measured resistivities are relatively independent of the method of measurement; the general trend of increasing resistivity with particle size should be noted.

A complete set of measurements by Smith-Rose [25] on a variety of soils from different depths, with different moisture contents, and measured at various frequencies, shows that at the lower frequencies (i.e. between 1 and 100 kHz) the conductivity of soils is essentially independent of frequency. At higher frequencies the conductivity rises and the increase in conductivity is generally greatest for the most poorly conducting samples. The results of his laboratory measurements on soil samples taken from various parts of England are illustrated in Table 18 and profiles of conductivity with depth

TABLE 14. Resistivities of igneous and metamorphic rocks (after W. M. Telford et al. [22])

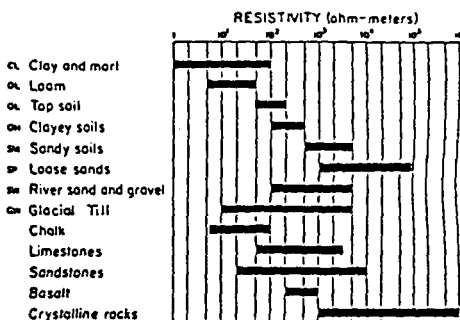
Rock type	Resistivity range (Ωm)
Granite	$3 \times 10^4 - 10^6$
Granite porphyry	4.5×10^3 (wet) - 1.3×10^4 (dry)
Feldspar porphyry	4×10^4 (wet)
Albite	3×10^4 (wet) - 3.3×10^5 (dry)
Syenite	$10^2 - 10^4$
Diorite	$10^2 - 10^4$
Diorite porphyry	1.9×10^3 (wet) - 2.8×10^4 (dry)
Porphyrite	10^{-5} - 10^4 (wet) - 3.3×10^3 (dry)
Carbonatized porphyry	2.5×10^3 (wet) - 6×10^4 (dry)
Quartz porphyry	$3 \times 10^2 - 9 \times 10^3$
Quartz diorite	$2 \times 10^2 - 2 \times 10^4$ (wet) - 1.8×10^4 (dry)
Porphyry (various)	$60 - 10^4$
Dacite	2×10^4 (wet)
Andesite	4.5×10^3 (wet) - 1.7×10^4 (dry)
Diabase porphyry	10^3 (wet) - 1.7×10^4 (dry)
Diabase (various)	$20 - 5 \times 10^3$
Lavas	$10^2 - 5 \times 10^4$
Gabbro	$10^2 - 10^4$
Basalt	$10 - 1.3 \times 10^2$ (dry)
Olivine norite	$10^2 - 6 \times 10^4$ (wet)
Peridotite	3×10^4 (wet) - 6.5×10^3 (dry)
Hornfels	8×10^2 (wet) - 6×10^3 (dry)
Schists (calcareous and mica)	$20 - 10^4$
Tuffs	2×10^3 (wet) - 10^4 (dry)
Graphite schist	$10 - 10^2$
Slates (various)	$6 \times 10^2 - 4 \times 10^3$
Gneiss (various)	6.8×10^4 (wet) - 3×10^4 (dry)
Marble	$10^2 - 2.5 \times 10^3$ (dry)
Skarn	2.5×10^3 (wet) - 2.5×10^4 (dry)
Quartzites (various)	$10 - 2 \times 10^4$

TABLE 15. Resistivities of sediments (after W. M. Telford et al. [22])

Rock type	Resistivity range (Ω m)
Consolidated shales	$20-2 \times 10^3$
Argillites	$10-8 \times 10^3$
Conglomerates	$2 \times 10^3-10^4$
Sandstones	$1-6.4 \times 10^4$
Limestones	$50-10^7$
Dolomite	$3.5 \times 10^3-5 \times 10^3$
Unconsolidated wet clay	20
Marls	3-70
Clays	1-100
Alluvium and sands	10-800
Oil sands	4-800

from these measurements are shown in Figure 12. It is seen from Table 18 that the soil type varies rapidly with depth, as does the soil moisture content; both of these influence the conductivity profiles. Smith-Rose concludes that clays have the highest conductivities, greater than 10 millimhos per meter, loams and chalks are of the order of 10 millimhos per meter and sandy or gritty soils are appreciably less. He also points out that a diurnal temperature range of 20°C at the surface of the soil represents a temperature change of approximately 1.4°C at a depth of one foot. Measurements made on a soil sample at 1.2 MHz as a function of moisture content by weight show a conductivity that increases approximately as the square of the moisture content.

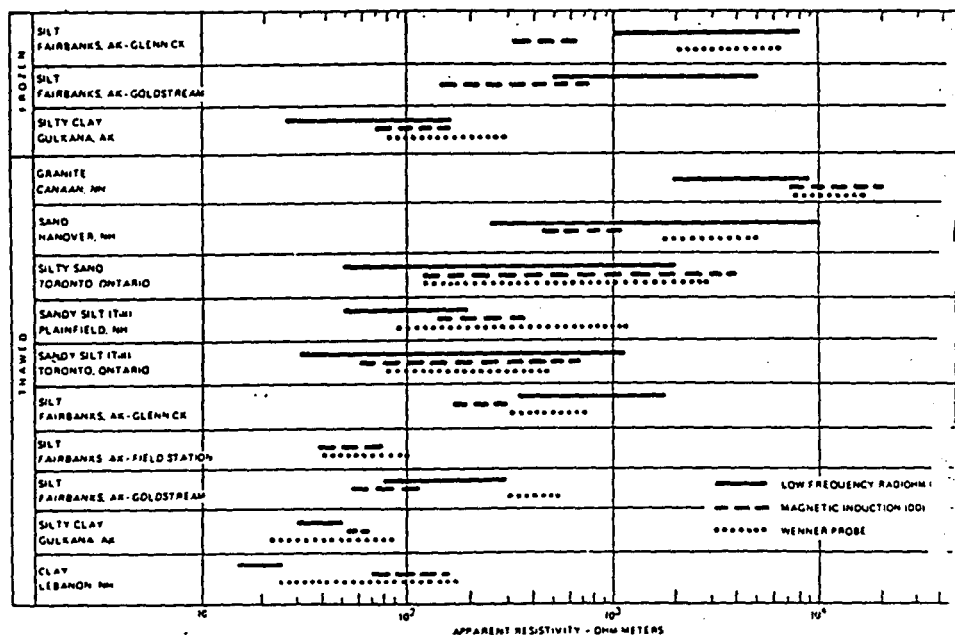
TABLE 16. Resistivity ranges for various terrain materials (after Cuiley et al. [23])



V. 2. Tropical Humid Zones [26]

The examples given above have been taken from temperate zone soils and seem to be fairly representative. In the case of soil occurring in tropical humid climates (annual temperatures of the order of 25°C ; annual precipitation of greater than a meter, generally falling during a part of the year) the weathering can be very deep. Indeed, unweathered parent rock may not occur until 30 meters flat country, 60 meters in hilly country and in rare occasions dept.

TABLE 17. Apparent resistivity data obtained for various material types using three measuring techniques (after Sellman et al. [24])



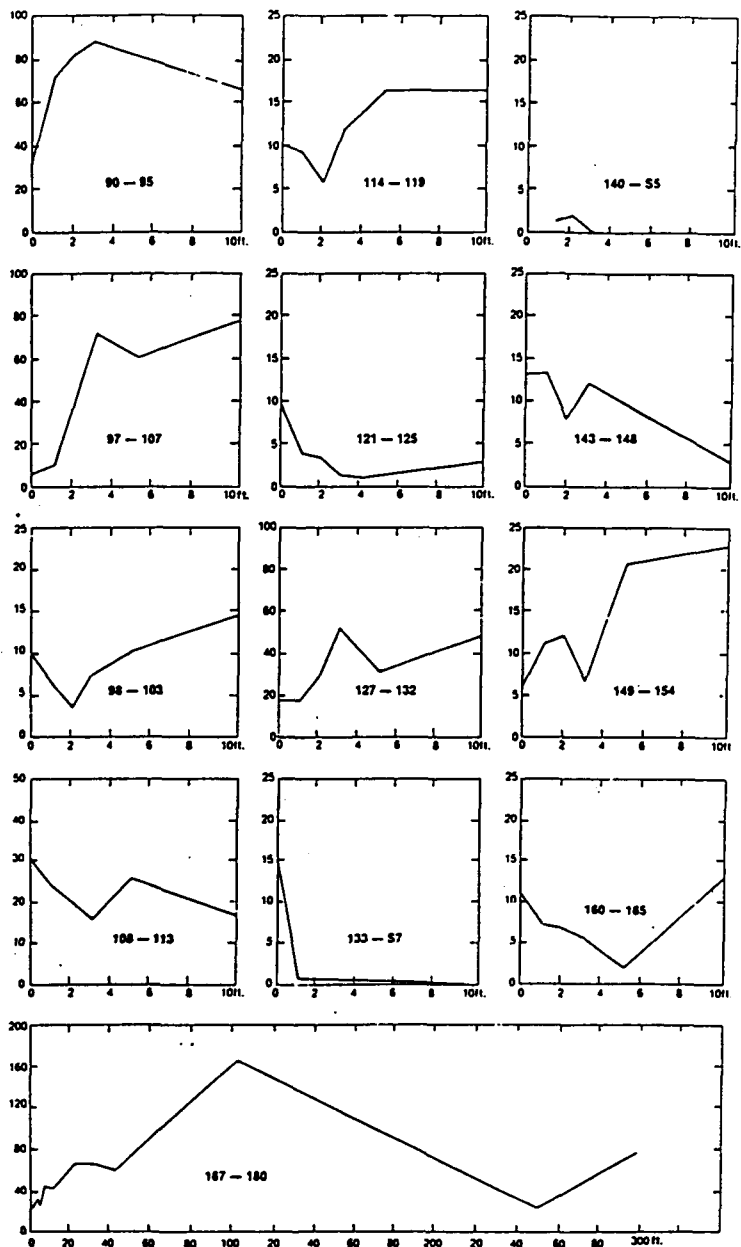


FIGURE 12. Plots of conductivity (mmhos/m) versus depth (feet) for data in Table 18.

TABLE 18. Measurements of samples of soil taken from different depths at various sites (after R. L. Smith-Rose [25])

Sample No.	Site	Geological classification	Depth	Description of sample	Moisture content	Conductivity at 20°C	
						1 kHz	100 kHz
			ft.		per cent		
90	Rugby Radio Station (1)	Lower lias	Surface	Dark fibrous loam	60	33	38
92			1	Loam and clay	33	72	78
93			2	Clay and sand	26	83	89
94			3	Blue clay	25	89	100
95			10	Blue clay	23	67	72
97	Rugby Radio Station (2)	Lower lias	Surface	Loam	22	6.1	7.2
104			1	Loam and clay	13	9.4	9.4
105			3	Blue clay	27	72	83
106			5	Clay and sand	21	61	61
107			10	Blue clay	25	78	89
98	Baldock, Herts (P.O. Receiving Station)	Chalk	Surface	Fibrous loam	21	9.4	10.0
99			1	Chalky loam	21	6.1	6.1
100			2	Chalk	24	8.1	2.9
101			3	Chalk	27	7.2	7.8
102			5	Chalk	26	10.1	15.6
103			10	Chalk	27	14.4	15.6
108	Tatsfield, Kent (B.B.C. Receiving Station)	Upper greensand	Surface	Fibrous loam	37	30	38
109			1	Brown, sandy clay	11	24	27
110			2	Brown sand	15	20	22
111			3	Light brown sand	13	15.6	10.7
112			5	Light brown sand	20	26	26
113			10	Yellow sand	15	16.7	19
114	Brookmans Park, Herts (London Regional Station)	London clay	Surface	Fibrous loam	19	10.0	10.6
115			1	Stony loam	18	9.4	10.0
116			2	Light sandy clay	22	5.6	7.2
117			3	Sandy clay	22	12.2	13.3
118			5	Sandy clay	21	16.7	18.8
119			8 to 10	Clay and shingle	10	16.7	17.8
121	Daventry Northants	Upper lias	Surface	Fibrous loam	28	9.4	10.6
122			1	Sandy loam	16	3.8	3.8
123			2	Brown sand	14	3.2	3.2
124			3	Brown sand	5.0	1.1	1.2
126			5	Sand and sandstone	8.5	0.8	1.0
125			10	Sand and sandstone	24	2.9	3.7
127	Washford Cross, Somerset (West Regional Station)	Red Marls	Surface	Reddish-brown loam	23	16.7	18.9
128			1	Reddish-brown clay	20	16.7	18.9
130			2	Reddish-brown clay	18	28.9	31
129			3	Reddish-brown clay	21	52	54
131			5	Reddish-brown clay	19	31	34
132			10	Reddish-brown clay	15	48	50
133	Brendon Hills, Somerset	Devonian	Surface	Black fibrous loam	210	14.4	16.7
134			1	Loam and slate	9.0	0.3	0.3
135			2	Loam and slate	9.0	0.2	0.2
136			3	Loam and slate	8.0	0.1	0.1
137			5	Loam and slate	5.5	0.0	0.0
S.7			10	Slate	—	0.0	0.0
140	Merrivale, Dartmoor, Devon	Granite	1	Gritty loam	18	1.3	1.3
141			2	Gritty loam	13	1.6	1.6
S.1			3 to 10	Granite	—	0.0	0.1
S.2			3 to 10	Granite	—	0.0	0.1
S.3			3 to 10	Granite	—	0.0	0.0
139	Dousland, Dartmoor, Devon	Devonian	Surface	Loam	47	6.1	6.7
142			1	Dark brown loam	41	2.7	2.7
S.4			Below 1	Slate	—	0.0	0.0
S.6			Below 1	Granite	—	0.0	0.0
143			Surface	Fibrous loam	130	13.3	17.8
144	Moorside, Edge, Yorks (North Regional Station)	Millstone grit	1	Dark grey clay	60	13.3	15.6
145			2	Dark grey clay	35	7.8	10.6
146			3	Dark grey clay	39	12.2	16.7
147			5	Dark grey clay	19	9.4	11.1
148			10	Yellow and grey clay	15	3.4	3.7
149	Westerglen, Falkirk (Scottish Regional Station)	Boulder clay	Surface	Fibrous loam	38	6.1	7.2
150			1	Fibrous loam	30	11.1	11.1
151			2	Clay and loam	19	12.2	12.2
152			3	Dark grit and clay	18	6.7	7.8
153			5	Dark grit and clay	18	21	24
154			10	Dark grit and clay	15	23	26

(continued over)

TABLE 18. (concluded)

Sample No.	Site	Geological classification	Depth	Description of sample	Moisture content	Conductivity at 20°C	
						Millimho/m	
			ft.		per cent	1 kHz	100 kHz
160	Teddington, Middlesex (N.P.L.)	London clay	Surface	Fibrous loam	26	11.1	12.2
161			1	Sandy loam	20	7.2	7.8
162			2	Sandy loam	13	6.7	7.2
163			3	Fine gravel	6.5	5.6	6.7
164			5	Coarse gravel	2.9	1.9	2.0
166			7	Fine sand	2.6	1.6	1.6
165			10	Sand and shingle	20	13.3	15.6
167	Wychbold, Droitwich (Midland Regional Station)	Red Marls	1	Red clay and loam	15	16.7	20
168			2	Red clay	13	34	32
169			3	Red clay	14	23	24
170			5	Red clay and stones	15	43	51
171			10	Red clay and stones	21	41	44
172			20	Red clay suspension	31	67	78
173			30	Red clay suspension	41	67	78
174			40	Red clay suspension	25	61	72
175			50	Red clay	27	78	83
176			100	Grey clay and salt	28	177	233
177			150	Red clay and salt	27	—	—
178			200	Red clay and salt	24	—	—
179			250	Red clay and salt	22	24	26
180			300	Red clay and salt	31	73	89

of 250 meters have been observed. The resulting soils are red to yellow, soft with a high clay content, and with a specific gravity approximately one-half of that of the parent rock. Compared with the soils of temperate climates they are thick, humus poor, permeable, and have a high clay-silt ratio. Although the clay content is high this is somewhat compensated for by the fact that in well drained soils the clays are kaolinitic and the cation exchange capacity is less than in temperate zones (see Table 11). Iron-rich concretionary horizons called laterites form as a result of reprecipitation. These are hard, permeable, and if dry are usually very resistive.

The nature and the extent of the soil formation is a function of the rock type, texture, jointing, surface relief, vegetation, water-table, and micro-climate. For example, the relative absence of silt is due to the fact that fine-grained rocks weather faster than coarse-grained rocks; the abundance of clay results from the fact that it is a stable mineral.

An excellent series of measurements of the electrical properties of the weathered zones in tropical climates has been carried out by Palacky and Kadekaru in Brazil [27]. The measurements were done

with conventional resistivity techniques and many soundings were taken. The results gave good agreement with a three-layered earth model and are summarized in Table 19. In general a resistive soil relatively thin, is situated on top of a thick quite conductive weathered zone, in turn situated on the relatively resistive parent rock.

V. 3. Tropical Arid Zones

It is in tropical arid zones that the most conductive soils are encountered. As for humid zones the weathered layer can be many tens of meters in thickness, however in arid climates this material often contains a high salt concentration due to evaporation. Whereas the surface materials are usually dry, possibly lateritic, and resistive, deeper material may approach resistivities of the order of 1 ohm-meter. Furthermore substantial lateral variations of resistivity are not uncommon.

In the humid tropical zones referred to above the conductivity of the intermediate zone was due to the presence of an abundance of colloidal particles with moderate ion exchange capacities; abundant rainfall means that drainage patterns are well established and soil salts have long since been leached into major river channels and

TABLE 19. Tropical resistivity profiles (after Palacky and Kadekaru [27])

	Site	Soil		Weathered Zone		Bedrock	
		Thickness (meters)	Resistivity (ohm-meters)	Thickness (meters)	Resistivity (ohm-meters)	Type	Resistivity (ohm-meters)
Humid Tropical	Nova Lima	5-10	2000-15000	20-80	50-100	schists	∞
	Canabrava	5	90	3-18	200-400	granodiorite	∞
		5	360	12-30	15-27	basalt	∞
	Santa Fé	3	11000-18000	35	3-12	ultrabasic	∞
	Quatipuro	0-1	200	15-35	80-100	dunite	∞
Arid Tropical			300-700	3-8	10-30	phyllites	∞
					35-20	serpentinites	> 300
	Santa Luz	-		3.5	65	granite	910
		1	120	10-20	13	basic volcanic	∞
	Curuçá	-		6-10	20-35	amphibolites	1200
				15-20	80-130	gneiss	2000

TABLE 20. Diagnostic criteria for distinguishing between unaffected soil sites and encroaching and developed saline seeps (after Rhoades and Halvorson [28])

Site type	Salt content	Water content	Soil electrical conductivity
Unaffected	Low, increasing with depth	Low, increasing with depth	Low, increasing with depth
Encroaching saline seep	Low, increasing to a peak at a relatively shallow depth, then decreasing with further depth	Moist surface, becoming wet with depth	Intermediate, increasing to a peak at a relatively shallow depth, then decreasing with further depth
Developed saline seep	High, decreasing with depth	Relatively uniformly wet to the water table	High, decreasing with depth

thence into the oceans. In hot arid climates drainage patterns are poorly established and drainage basins may have no outlet to permanent streams. Salt-bearing waters drain from topographically high regions into lower areas where the water evaporates, leaving a high residual salt content, largely a reflection of the imperfect drainage channels.

The occasional addition of water might be due either to precipitation or irrigation. In the cultivated dry land soils of the northern Great Plains the near surface (plant root-zone) is leached with essentially pure water derived from rain and snow melt during the wet months. Rhoades and Halvorson [28] discuss how this results in a soil salinity concentration which increases with depth. However during the summer months when a saline water table in this climate is situated within approximately one meter from the surface an upwards flow of moisture is caused by evaporation at the soil surface and transpiration within the root zone. This upwards flow reverses the original leaching which transported salts downwards and causes them to ascend so that a concentration peak can form in the soil salinity profile. The soils can remain excessively wet as a

result of the hygroscopic nature of the salts and because this reduces the effective use of water by plants via evapo-transpiration. Rhoades defines such a condition as a "saline seep" and notes that it can form without the water table actually emerging at the surface. As this seep develops the peak in the soil salinity profile moves upwards until it finally appears at or near the surface as indicated in Table 20 which illustrates the various conditions that can exist in a region which sees seasonal precipitation followed by seasonal drought. Figure 13 illustrates vertical profiles of resistivity at depths as determined by an expanding Wenner array for the conditions described in Table 20.

For saline soils, the contribution to conductivity from cation concentration generally outweighs that from cation exchange capacity, and the conductivity is relatively independent of clay content.

The effect of weathered-zone salinity is probably at its maximum in western Australia where the zone is often 100 meters in thickness and has been known to exceed 300 meters. Hygroscopic salinity can maintain soil moisture at levels substantially above the water table. Resistivities of the order of 1 ohm-meter have been observed. Lateral variations result from the imperfect drainage pattern: changes in surface topography often show up as large changes in subsurface conductivity. A lateritic hard pan is not unusual in this environment. This layer is extremely resistive and complicates the complexity and difficulty of making conventional electrical measurements in this environment.

V. 4. Arctic Zones

Finally we turn our attention to arctic regions in which permafrost is a major consideration. It should be noted that the definition of permafrost requires only that the mean annual temperature of the ground be less than 0°C for several years; the definition is completely independent of the nature of the material, the amount of moisture, and indeed whether or not the moisture is from

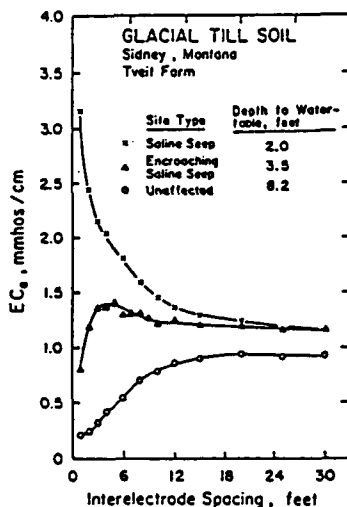


FIGURE 13. Relation between soil electrical conductivity, EC_e , and interelectrode spacing for a saline seep, an encroaching saline-seep site, and an unaffected site for glacial till-clay loam soil near Sidney, Montana (after Rhoades and Halvorson [28]).

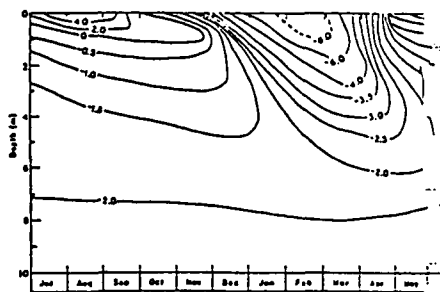


FIGURE 14. Vertical and temporal temperature variations - contour (after Arcone et al [29]).

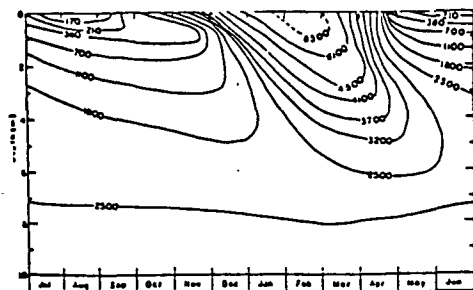


FIGURE 15. Computed vertical and temporal resistivity variations from data of Figure 14 - contoured in ohm-meters (after Arcone et al [29]).

In northern climates we find that mineralogy, porosity, free water content, and ionic concentration within the free water are important resistivity factors as they are for temperate zones. In permafrost regions we have the additional complexity that small changes in temperature of the ground near 0°C may exert a large influence on terrain conductivity. The effect depends on the nature of the material and also the moisture content since as we have seen earlier even at temperatures substantially below 0°C considerable moisture may remain unfrozen in the case of clays whereas in the case of coarser material virtually all will be frozen. In short, ice content is a complicated function of many variables.

As an example of electrical layering arising due to temperature only consider Figure 14 which illustrates annual variations in ground temperature recorded near Fairbanks, Alaska. The region is in a discontinuous permafrost zone and subsurface material consists primarily of perennially frozen organic silt containing a varying amount of ground ice. From laboratory measurements on saturated organic silt to determine the variation of electrical resistivity with temperature, a plot of electrical resistivity with depth and time was derived [23] and is shown in Figure 15. We see from this figure that a resistivity range of 50 to 1 can occur as a result of temperature changes alone.

In the discontinuous zone of permafrost subsurface temperatures are subtly influenced by many variables and even in ground which is

laterally uniform insofar as material type is concerned lateral temperature variations can bring about large changes in electrical resistivity as shown in Figure 16 where the survey traverse was taken over glacial lake basin sediments. The frozen areas outlined on the figure were derived from resistivity measurements and confirmed by drilling [24].

VI. SUMMARY

In this technical note we have discussed the concept of terrain conductivity, examined some of the properties of soils and rocks that affect their conductivity, and reviewed some measurements on typical terrain materials under a variety of climatic conditions.

To summarize, terrain conductivity is usually determined by one or more of the following parameters:

- (1) clay content, clay type
- (2) moisture profile with depth
- (3) moisture salinity
- (4) moisture temperature

Of these the most complex is usually the moisture profile, by which is meant the way in which (i) the porosity, (ii) the extent to which the pores are filled with water, and, (iii) the number, size and shape of interconnecting passages all vary with depth. The moisture profile is affected by material type (directly influencing porosity etc., or indirectly influencing permeability and water table location), topography (influencing the water table location), compaction (influencing porosity), and season (rates of precipitation, evaporation).

It is evident that many parameters may affect the ground conductivity. Fortunately at any given location relatively few are usually dominant however the survey interpreter should be aware of the possible alternatives. It is hoped that this brief treatment will be useful in assessing those parameters that are influential in the survey area and will thereby lead to a more accurate interpretation of survey data.

The bibliography gives a good indication of current interest in the electrical properties of rocks and soils and the curious reader will find much valuable material in the cited references.

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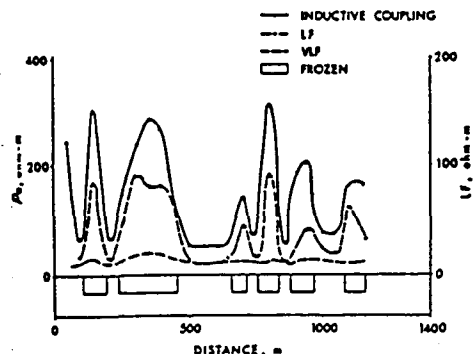


FIGURE 16. Results of the apparent resistivity measured with VLF (18.6 Hz), LF (375 kHz) radiohm and a magnetic induction instrument (EM31) over a section of shallow discontinuous permafrost (after P. Hoekstra [30]).

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APPENDIX G-6

FIELD SOIL GAS SAMPLING AND ANALYSIS



**FIELD OPERATION PLAN
WITH
QA/QC PROCEDURES
Tracer Research Corporation**

SUBMITTED BY:

11/30/89
Date

Wanda X. Bodle
Tracer Research Corporation



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SOIL GAS SAMPLING PROCEDURE

I. Probe Placement

- A) A clean probe (pipe) is removed from the "clean" storage tube on top of the van.
- B) The soil gas probe is placed in the jaws of hydraulic pusher/puller mechanism.
- C) A sampling point is put on the bottom of the probe.
- D) The hydraulic pushing mechanism is used to push the probe into the ground.
- E) If the pusher mechanism will not push the probe into the ground a sufficient depth for sampling, the hydraulic hammer is used to pound the probe into the ground.

II. Sample Extraction

- A) An adaptor (Figure 1) is put onto the top of the soil gas probe.
- B) The vacuum pump is hooked onto the adaptor.
- C) The vacuum pump is turned on and used to evacuate soil gas.
- D) Evacuation will be at least 30 seconds but never more than 5 minutes for samples having evacuation pressures less than 15 inches of mercury. Evacuation times will be at least 1 minute, but no more than 5 minutes for probes reading greater than 15 inches of mercury.
- E) Gauges on the vacuum pump are checked for inches of mercury.
 - 1. Gauge must read at least 2 inches of mercury less than maximum vacuum to be extracting sufficient soil gas to collect a valid sample.



III. Sample Collection

- A) With vacuum pump running, a hypodermic syringe needle is inserted through the silicone rubber and down into the stainless steel tubing of adaptor (Figure 1).
- B) Gas samples should only contact steel surfaces and never contact potentially sorbing materials (i.e., tubing, hose, pump diaphragm).
- C) The syringe is purged with soil gas then, without removing syringe needle from adaptor, a 2-10 mL soil gas sample is collected.
- D) The syringe and needle are removed from the adaptor and the end of the needle is capped.
- E) If necessary, a second 10 mL sample is collected using the same procedure.

IV. Deactivation of Sampling Apparatus

- A) The vacuum pump is turned off and unhooked from the adaptor.
- B) The adaptor is removed and stored with equipment to be cleaned.
- C) Using the hydraulic puller mechanism, the probe is removed from the ground.
- D) The probe is stored in the "dirty" probe tube on top of the van.
- E) The probe hole is backfilled, if required.

V. Log Book and U.S. EPA Field Sheet Notations For Sampling (Figures 2A-2D)

- A) Time (military notation)
- B) Sample number (use client's numbering system)
- C) Location (approximate description - i.e., street names)
- D) Sampling depth



- E) Evacuation time before sampling
- F) Inches of mercury on vacuum pump gauge
- G) Probe and adaptor numbers
- H) Number of sampling points used
- I) Observations (i.e., ground conditions, concrete, asphalt, soil appearance, surface water, odors, vegetation, etc.)
- J) Backfill procedure and materials, if used.

VI. Other Recordkeeping

- A) Client-provided data sheets are filled out, if required
- B) Sample location is marked on the site map

VII. Determination of Sampling Locations

- A) Initial sample locations will be determined by client (perhaps after consultation with TRC personnel) prior to start of job.
- B) Remaining sample locations may be determined by:
 - 1) Client
 - a) Entire job sampling locations set up on grid system.
 - b) Client decides location of remaining sample locations based on results of initial study, or
 - 2) Client and TRC Personnel
 - a) Client and TRC personnel decide location of remaining sample locations based on results of initial sample locations.



ANALYTICAL PROCEDURES

- I. Varian 3300 Gas Chromatograph
 - A) Equipped with Electron Capture Detectors (ECD), Flame Ionization Detectors (FID), Photo Ionization Detectors (PID) and/or Thermal Conductivity (TC) Detectors.
 - B) The chromatographic column used by TRC for the analysis of halocarbons is a 1/8" diameter packed column containing Alltech OV101. This nicely separates most of the tri-chloro and tetra-chloro compounds that are encountered in soil gas investigations. The di-chloro compounds tend to elute ahead of the tri-chloro and tetra-chloro compounds, thus creating no interference. In the event that assurance of the identity of a compound in any particular sample is needed, it will be analyzed on a SP-1000 column after the OV-101 analysis.
- II. Two Spectra Physics SP4270 Computing Integrators.

The integrators are used to plot the chromatogram and measure the size of the chromatographic peaks. The integrators compute and record the area of each peak. The peak areas are used directly in calculation of contaminant concentration.
- III. Chemical Standards From ChemServices, Inc. of Westchester, Pennsylvania.
 - A) TRC used analytical standards that are preanalyzed, of certified purities and lot numbered for quality control assurance. Each vial is marked with an expiration date. All analytical standards are the highest grade available. Certified purities are typically 99%.



- B) The Quality Assurance procedures used by ChemService were described by the Laboratory Supervisor, Dr. Lyle Phipper:
- 1) The primary measurement equipment at ChemServices, the analytical balance, is serviced by the Mettler Balance Company on an annual basis and recalibrated with NBS traceable weights.
 - 2) All chemicals purchased for use in making the standards are checked for purity by means of gas chromatography using a thermal conductivity detector. Their chemicals are purified as needed.
 - 3) The information on the purification and analysis of the standards is made available upon request for any item they ship when the item is identified by lot number. All standards and chemicals are shipped with their lot numbers printed on them. The standards used by TRC are made up in a two step dilution of the pure chemical furnished by ChemServices.

IV. Analytical Supplies

1. Sufficient 2 and 10 cc glass and Hamilton syringes so that none have to be reused without first being cleaned.
2. Disposable lab supplies, where appropriate.
3. Glassware to prepare aqueous standards.
4. Miscellaneous laboratory supplies.



QA/QC PROCEDURES

I. Standards

- A) A fresh standard is prepared each day. The standards are made by serial dilution.
- 1) First, a stock solution containing the standard in methanol is prepared at TRC offices in Tucson. The stock solution is prepared by pipetting the pure chemical into 250 mL of methanol in a volumetric flask at room temperature. The absolute mass is determined from the product of volume and density calculated at room temperature. Hamilton microliter syringes, with a manufacturer's stated accuracy of + or - 1%, are used for pipetting. Information on density is obtained from the CRC. Once the stock solution is prepared, typically in concentration range of 50-1000 mg/L, a working standard is prepared in water each day. The solute in the stock solution has a strong affinity to remain in methanol so there is no need to refrigerate the stock solution. Additionally, the solute tends not to biodegrade or volatilize out of the stock solution.
- 2) The working standards are prepared in 43 mL VOA septum vials by diluting the appropriate ug/L quantity of the standard solution into 43 mL of water.
- B) The standard water is analyzed for contamination before making the aqueous standard each day.
- C) The aqueous standard is prepared in a clean vial using the same syringe each day. The syringe should only be used for that standard.



- D) Final dilution of the calibration standards are made in water in a VOA vial having a Teflon coated septum cap instead of in a volumetric flask in order to have the standard in a container with no air exposure. The VOA bottle permits mixing of the standard solution and subsequent syringe sampling all day long without opening the bottle or exposing it to air. The measurement uncertainty inherent in the use of a VOA bottle instead of a volumetric flask is approximately + or - 1%.
- E) The aqueous standard will contain the compounds of interest in the range of 5 to 100 ug/L depending on the detectability of the individual components. The standard will be analyzed at least 3 times at the start of each day to determine the mean response factor (RF) for each component (Figure 3). The standard will be injected again after every fifth sample to check detector response and chromatographic performance of the instrument throughout the day.
- F) The RF allows conversion of peak areas into concentrations for the contaminants of interest. The RF used is changed if the standard response varies 25%. If the standard injections vary by more than 25% the standard injections are repeated. If the mean of the two standard injections represents greater than 25% difference then a third standard is injected and a new RF is calculated from the three standard injections. A new data sheet is started with the new RF's and calibration data.

$$\% \text{ difference} = \frac{A \text{ area} - B \text{ area}}{A \text{ area}}$$

Where A = mean peak area of standard injection from first calibration

B = peak area of subsequent standard injection



- G) The low ug/L aqueous standards that are made fresh daily need not be refrigerated during the day because they do not change significantly in a 24 hour period. On numerous occasions the unrefrigerated 24 hour old standards have been compared with fresh standards and no difference has been measurable. If the standards were made at high ppm levels in water, the problem of volatilization would probably be more pronounced in the absence of refrigeration.
- H) Primary standards are kept in the hotel room when on a project.
- I) A client may provide analytical standards for additional calibration and verification.

II. Syringe Blanks

- A) Each uL syringe is blanked before use.
- B) 2 cc (glass) syringes will each be blanked if ambient air concentrations are elevated (greater than or equal to 0.01 ug/L) for components of interest.
- C) If ambient air concentrations are <0.01 ug/L for components of interest, a representative sample of at least two syringes are blanked at the start of each day. If representative syringes have no detectable contamination remaining syringes need not be blanked. If any of representative syringes show contamination, all 2 cc syringes must be blanked prior to use.
- D) Syringe blanks are run with air or nitrogen.
- E) If it is necessary for any syringe to be used again before cleaning, it is blanked prior to its second use.



III. System Blanks

- A) System blanks are ambient air drawn through the probe and complete sampling apparatus (probe adaptor and 10 cc syringe) and complete sampling apparatus and analyzed by the same procedure as a soil gas sample. The probe is above the ground.
- B) One system blank is run at the start of each day and compared to a concurrently sampled air analyses.
- C) A system blank will all be run before reusing any sampling system component.

IV. Ambient Air Samples

- A) Ambient air samples are collected and analyzed a minimum of three times daily to monitor safety of the work environment and to establish site background concentrations, if any, for contaminants of interest.
- B) All ambient air samples shall be documented (Figure 3).

V. Samples

- A) All unknown samples will be analyzed at least twice.
- B) More unknown samples will be run until reproducibility is within 25%, computed as follows:

$$\text{Difference} = \frac{A - B}{(A + B)/2}$$

Where: A is first measurement result

B is second measurement result

If the difference is greater than .25, a subsequent sample will be run until two measurements are made that have a difference of .25 or less. Those two measurements will be used in the final calculation for that sample.

- C) The injection volume should be adjusted so that mass of analyte is as near as possible to that which is



contained in the standard, at least within a factor of 10.

- D) Whenever possible the attenuation for unknown samples is kept constant through the day (so as to provide a visual check of integrations).
- E) A water plug must be used as a gas seal in uL syringes
- F) A seal must be established between syringes when sub-sampling
- G) At very high concentrations air dilutions are acceptable once concentration of contaminants in air have been established.
- H) All sample analysis are documented (Figure 3).
- I) Separate data sheet are used if chromatographic conditions change
- J) Everything is labeled in ug/L, mg/L, etc. PPM and PPB notations are to be avoided.

VI. Daily System Preparation (Figure 4).

- A) Integrators parameters are initialized
 - 1. Pt. evaluation
 - 2. Attenuation
 - 3. Peak markers
 - 4. Auto zero
 - 5. Baseline offset (min. 10% of full scale)
- B) The baseline is checked for drift, noise, etc.
- C) System parameters are set.
 - 1. Gas flows (Note: N₂, air, H₂ tank pressure on Page 1 of chromatograms).
 - 2. Temperatures
 - a) Injector
 - b) Column
 - c) Detector



- D) After last analysis of the day conditioned septa are rotated into injection ports used during the day and replaced with fresh septa.
- E) Column and injector temperatures are run up to bake out residual contamination
- F) Syringes are cleaned each day
 - 1. 2 and 10 cc syringes are cleaned with Alconox or equivalent detergent and brush
 - 2. uL syringes are cleaned daily with IPA or MeOH and purged with N₂. Syringe Kleen is used to remove metal deposits in the barrel.
 - 3. Syringes are baked out overnight in the oven of the gas chromatograph at a minimum temperature of 60°C.

VII. Sample Splits

If desired, TRC's clients or any party, with the approval of TRC's client, may use sample splits to verify TRC's soil gas or groundwater sampling results.

- A) Sample splits may be collected in two valve, flow through-type all-glass or internally electroplated stainless steel containers for analysis within 10 days of collection.
 - 1. Flow through sample collection bottles should be cleaned by purging with nitrogen at 100°C for at least 30 minutes. Once clean, the bottles should be stored filled with nitrogen at ambient pressure.
 - 2. Sample bottles are filled by placing them in the sample stream between the probe and the vacuum pump. Five sample bottle volumes should be drawn through the container before the final sample is collected. The sample should be at ambient pressure.



- B) Sample splits can be provided in 10 cc glass syringes for immediate analysis in the field by the party requesting the sample splits.
- C) Splits of the aqueous standards or the methanol standards used by TRC for instrument calibration may be analyzed by the party requesting sample splits.



Figures 1 through 4

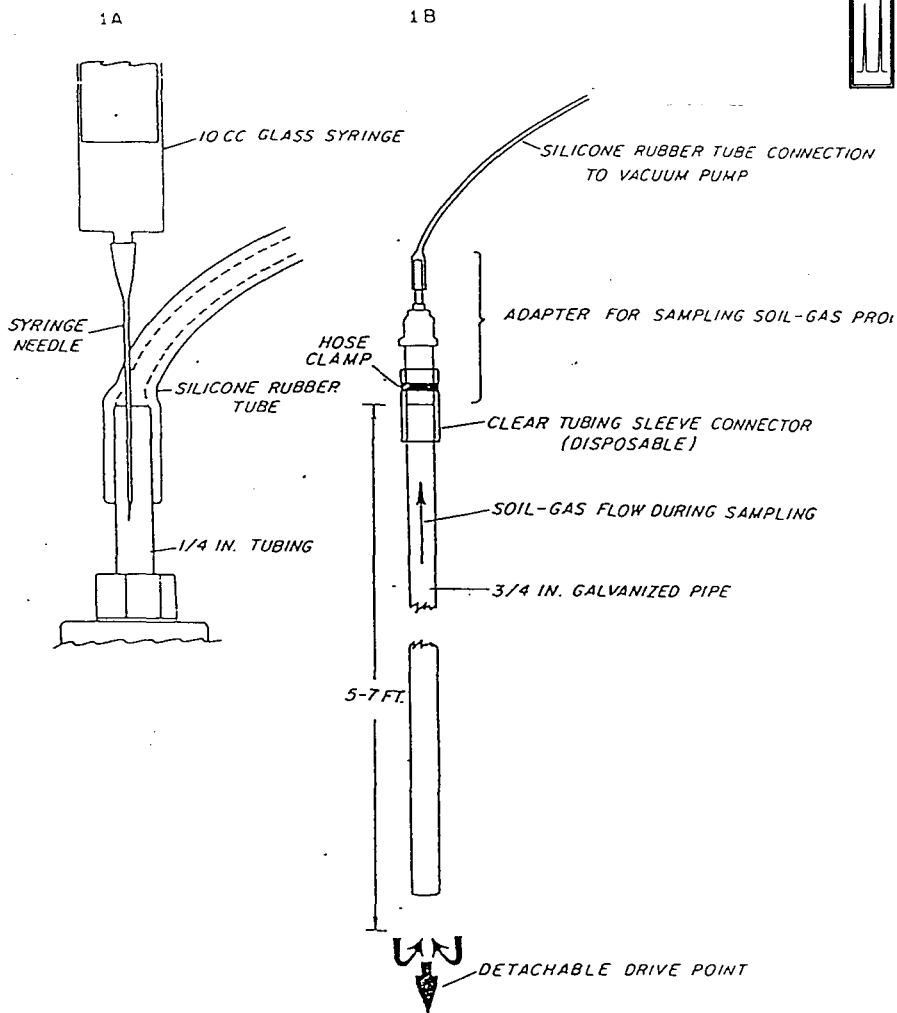


FIGURE 1. SAMPLING APPARATUS

- 1A. CLOSE-UP OF SYRINGE SOIL GAS SAMPLING THROUGH EVACUATION LINE
- 1B. DIAGRAM OF SOIL GAS SAMPLING PROBE WITH ADAPTOR FOR SAMPLING AND EVACUATION OF THE PROBE AFTER IT IS DRIVEN INTO THE GROUND



SOIL GAS INVESTIGATION BACKGROUND INFORMATION

SITE NAME: DAVIDSON CHEMICAL
 LOCATION: 14100 WEST AVENUE N. LANTHORN, SOUTH DAKOTA
 DATES OF INVESTIGATION: 2/16 - 2/18/88
 CLIENT NAME & ADDRESS: BRANDENBURG CONSULTANTS
602 HARRISON ROAD
WHEATWORTH SD 57863
 FIELD REPRESENTATIVE(S) FOR CLIENT: JOSEPH PANDOLFI
 PERSON TO WHOM REPORT AND QUESTIONS
 SHOULD BE DIRECTED: SARAH WHITTEL
 PHONE: (783) 972-1003 EXT. 5
 CREW: CHEMIST S. KUTER GEOLOGIST M. FIVRON
 REPORT TO INCLUDE (CIRCLE):
 QA/QC-PROCEDURES-DATA ONLY or FULL REPORT WITH CONTOUR MAPS
 AND INTERPRETATION

PURPOSE OF INVESTIGATION

DETERMINE EXTENT OF CONTAMINATION FROM STORAGE TANK FILL

TARGET VOCs

<u>TOL</u>		
<u>TCE</u>		
<u>ICE</u>		

GROUNDWATER INFORMATION:

DEPTH TO WATER: 12-16' DIRECTION: NORTHEAST

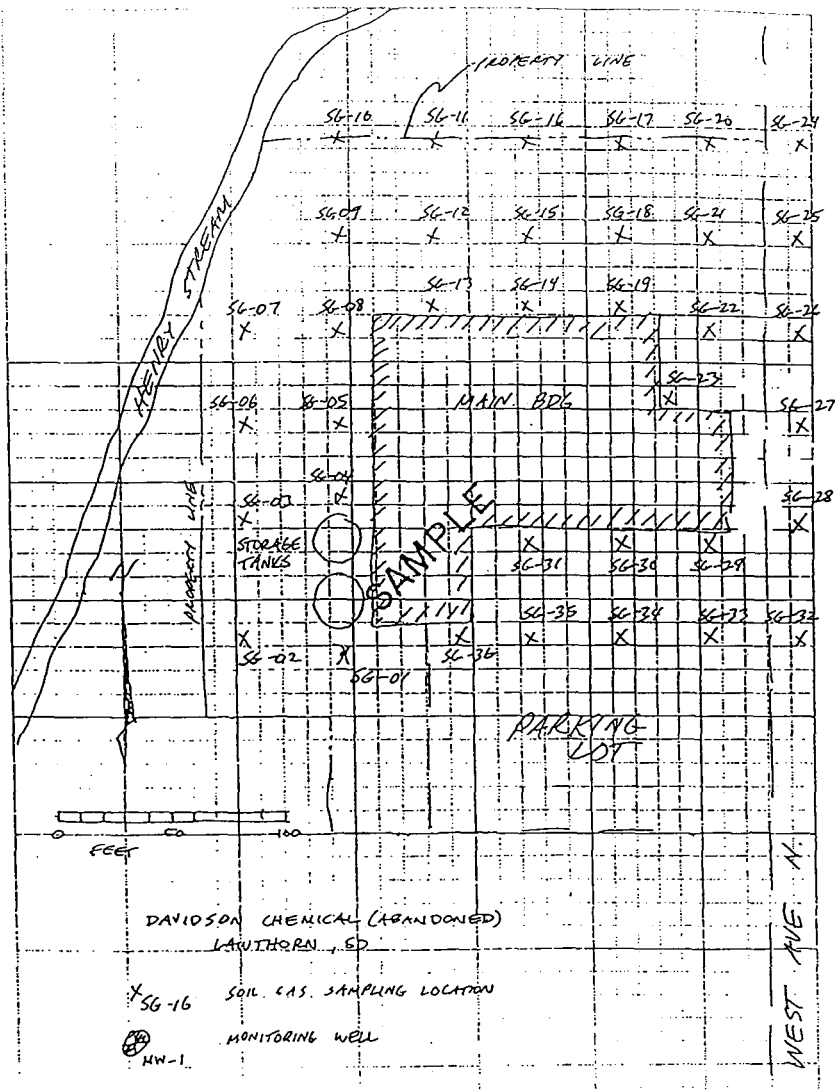
SOURCES OF CONTAMINATION

COMPANY USED SOLVENTS IN PHOTO-ETCHING PROCESS IN MANUFACTURE
OF ELECTRONIC CIRCUIT BOARDS. STORAGE TANK CRACKED AND LEAKED
FROM APPROX 1971-1982 WHEN COMPANY SHUT DOWN. SOURCE WAS
REMOVED IN 1982.

GEOLOGIC SETTING:(e.g. soil type, subsurface geology, etc.)

LOESS & GLACIAL TILL (~10'); FRACTURED BASEMENT @ 10'

FIGURE 2A. FIELD LOGBOOK - BACKGROUND INFORMATION



SITE MAPS TO INCLUDE: SITE NAME, SCALE, NORTH ARROW, SOIL GAS LOCATIONS AND NUMBERS, CULTURAL AND NATURAL FEATURES TO IDENTIFY SITE

FIGURE 2B. FIELD LOGBOOK - SITE MAP

DATE : 2-16-85
 LOCATION : DAVIDSON CHEMICAL
 CLIENT : BRANDENBURG

Chemist : S. KUPPER
 Weather : 12°; CLEAR & COLD

Geologist : M. FAVERON

FIELD HOURS

*Time on site : 730	Lunch hours : 1
*Time off site : 1730	Downtime hours : 0
Hours on site (P - A) : 10	Standby hours : 0

DECONTAMINATION

Probe Decontamination	Syringe Decontamination
Time start : 1745	Time start : 2000
Time end : 1800	Time end : 2030
Total hours : 1/4	Total hours : 1/2
Verified by geologist : <i>M. Favaron</i>	Verified by chemist : <i>S. Kupper</i>

SAMPLE

DAILY SUMMARY

Calibration	Sampling	Analysis
Time start : 730	Max vacuum : 23 in Hg	Total system
Time end : 830	Probes used : 18	blanks : 1
Total hours : 1	Points used : 20	Total air
	Soil gas samples collected : 18	samples : 3
	Water samples collected : 0	
Field data and gas standards checked by <i>M. Favaron</i>		
Data checking hours : 1 1/2		

- * - Downtime includes time spent repairing sampling & analytic equipment; note times and explanation on following field data pages
- ** - Standby includes time available for sampling but waiting for client; note times and explanation on following field data pages

FIGURE 2C. FIELD LOGBOOK - DAILY SUMMARY

DATE: 2-16-68
LOCATION: DAVIDSON CHEMICAL

CLIENT: BRANDENBURG

[illegible]

FIELD DATA SHEET

Tracer Research Corporation

DATE 2/16/89SITE NAME DAVIDSON CHEMICALLOCATION LAWTHORN, S. DAKOTAPAGE 1 OF 5CLIENT BRANDENBURG CONSULTANTS

CONFIDENTIAL

	① FI13				TEA				TCE				PCE				
standard conc.	② 10		ug/l		5		ug/l		10		ug/l		5		ug/l		
response from Sui injection	1	9531	③	area	1	9297	area	1	11488	area	1	18060	area	1	18060	area	
	2	10368		area	2	9167	area	2	11141	area	2	17572	area	2	17572	area	
	3	10719		area	3	8887	area	3	11265	area	3	12985	area	3	12985	area	
RFs for this sheet				④	4.90 x 10 ⁻¹⁵		q/area	2.74 x 10 ⁻¹⁵		q/area	4.43 x 10 ⁻¹⁵		q/area	1.54 x 10 ⁻¹⁵		q/area	
sample	⑤ inc	⑥ amt	⑦ area	⑧ ug/l	⑨ mean	area	ug/l	mean	area	ug/l	mean	area	ug/l	mean	area	ug/l	mean
N ₂ BLANK	805	1000	<1000	40.005		<300	40.008		4000	40.004		<300	40.0005		<300	40.0005	
AIR SAMPLE	810	1000	2000 ^E	0.01		4702	0.01		41000	40.004		5000 ^B	0.001		5000 ^B	0.001	
SYSTEM BLANK	845	1000	2000 ^B	0.01		5560	0.02		41000	40.004		4972	0.008		4972	0.008	
SG1-5'	941	1000	<1000	40.005	40005	5400	0.02	0.02	351625	2	200	26133	0.04	7	200	26133	0.04
SG1-5'	947	1000	<1000	40.005		58745	0.02		410552	2	200	25134	0.04		25134	0.04	
WS-18	955	1	397	2	72	<300	40.8	40.8	40528	200	200	4300	40.5	7	200	4300	40.5
WS-18	1003	1	392	2		<300	40.8		44715	200	200	<300	40.5		<300	40.5	

FIGURE 3
EXPLANATION OF FIELD DATA SHEET

- Name of compound.
- Concentration of analyte in calibration standards.
- Peak areas obtained from standard injections during calibration.
- Response factor (RF) for compound obtained from three calibration runs. The RFs are used for calculation of actual concentrations and are included on each data sheet.
- Hydrogen flame verifies decontamination of syringes and analytical equipment.
- Air sample gives ambient concentrations for comparison with system blanks.
- System blank verifies decontamination of sampling equipment.
- Sample ID Number. 805-810: 1000 g test sample 1 taken 2 feet down. 941-955: 1000 g test sample.
- Time of analysis identifies the chromatogram from which the data was taken.
- Amount of sample injected - information needed for the calculation of concentration.
- Peak area - raw number produced by the peak integrator that is proportional to the mass of analyte in the sample.
- Actual concentration present in the sample of soil gas or water rounded to one significant figure.
- Signature line for analyst and person that sheets data.

Notations: RF response factor
I interference with adjacent peaks
NA not analysed
E estimated peak area

Analyzed by S. Dupper ⑬
Checked by M. Kuen
Gas std checked by M. Kuen

DATE "12/18/85"
 TIME "07:15:46"
 FI= 1. FE= 1. MH= 0.
 PRESS "ENTER" TO SKIP ENTRY
 FILE NAME=""
 TIME FUNCTION VALUE
 TT= .01 TF= " AZ TV= 1
 TT= .01 TF= " PH TV= 1
 TT=

METHOD NUMBER: MH= 0

END OF DIALOG

OF=10

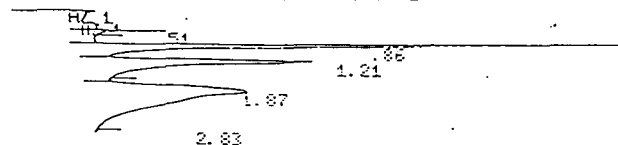
PT=100

AT= 8

Operator D-EDWARDS Date 12/18/85
 Column No. 62 Length 60' Dia. 1/8"
 Coating OV-101 Conc 0.5%
 Support Chromal W Mesh 80/100
 Column TEMP: Iso 100 °C Program
 Inlet (1) °C hold min Final (1) °C hold min
 CARRIER GAS N₂ Rate 40 ml/min
 Pressure: Inlet 50 Outlet 21
 Hydrogen 30 ml/min Air 300 ml/min
 DETECTOR: EC TC FID PID
 Baseline 17 mV Range 1 Attn 1
 Standard Composition (50) BACULIF (50) TOLUENE
(1226) ETHYL AROMATIC KEROSENE
100% CH₄

CHANNEL A INJECT 12/18/85 07:15:46

2.76
 CHANNEL A INJECT 12/18/85 07:23:10

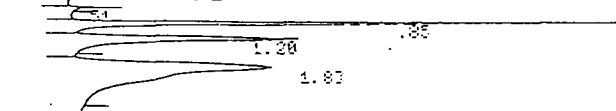


12/18/85 07:23:10 CH= "R" PS= 1.

FILE 1. METHOD 0. RUN 1 INDEX 1

PEAK#	AREA%	RT	AREA BC
1	1.167	0.51	1817 01
2	27.82	0.86	43300 08
3	20.802	1.21	32376 05
4	50.21	1.87	78148 01
TOTAL	100		155641

CHANNEL A INJECT 12/18/85 07:25:38
 12 1



CHANNEL A INJECT 12/18/85 07:59:18



FIGURE 4. CHROMATOGRAM DOCUMENTATION

APPENDIX G-7

CALIBRATION AND MAINTENANCE OF THE HNU PHOTOIONIZER

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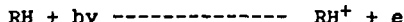
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 - 1.0 OPERATION PRINCIPLE
 - 2.0 INSTRUMENT SENSITIVITY AND CALIBRATION
 - 3.0 INSTRUMENT SPECIFICATIONS
- II. OPERATIONAL PROCEDURE
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 - 2.1.2 CALIBRATION FREQUENCY
 - 2.1.3 CALIBRATION PROCEDURE
 - 2.2 SAMPLE MEASUREMENTS
- III. MAINTENANCE AND TROUBLE SHOOTING
 - 1.0 BATTERY RECHARGING
 - 2.0 GENERAL FAULTS DETERMINATION AND CORRECTION
 - 3.0 SPECIFIC FAULTS

OPERATION PROCEDURE FOR
HNU MODEL PI 101
PHOTOIONIZATION ANALYZER

I. INTRODUCTION

1.0 Operation Principle

The HNU Model 101 photoionization detector has been designed to measure the concentration of trace gases in many industrial or plant atmospheres. The instrument has similar capabilities outdoors. The analyzer employs the principle of photoionization for detection. This process is termed photoionization because the absorption of ultraviolet light (a photon) by a molecule leads to ionization via:



where RH = trace gas

h ν = a photon with an energy greater than or equal to an ionization potential of RH.

The sensor consists of a sealed ultraviolet light source that emits photons which are energetic enough to ionize many trace species (particularly organics), but do not ionize the major components of air such as O₂, N₂, CO, CO₂ or H₂O. A chamber adjacent to the ultraviolet light source contains a pair of electrodes. When a positive potential is applied to one electrode, the field created drives any ions, formed by absorption of UV light, to the collector electrode where the current (proportional to concentration) is measured. The useful range of the instrument is from a fraction of a ppm to about 2,000 ppm.

2.0 Instrument Sensitivity and Calibration

The instrument responds to atmospheric compounds with ionization potentials equal to or less than the ionization energy of the UV light source. If a compound in air has an ionization potential greater than the energy source of the lamp, it will not be detected. Table 1 presents organic and inorganic compounds and the light sources that should be used to detect each compound. The instrument is capable of using 1 of the 3 light sources - 9.5, 10.2, and 11.7 ev lamps. In addition, not all compounds respond equally to each light sources and thus they vary in their sensitivity to ionization. As a result of varying sensitivities to photoionization, the response given by the instrument may or may not reflect the actual atmospheric concentration of the compound being

detected. Table 2 represents the relative sensitivities for various gases relative to a 10.2 ev light source. Use this table to determine the approximate response of the instrument to a compound of interest, and to select the appropriate light (lamp) source.

There are two types of operations that are used for calibration. For Type 1 Operation, a non-regulatory (or non-target) compound such as isobutylene is used for calibration. In this case, the instrument reading is reported in terms relative to the calibration compound used for calibration. For the type 2 operation, the target compound or compounds are used for calibration. As a result, the instrument is calibrated to respond directly in ppm by volume of the target compound(s).

3.0 Instrument Specifications

3.1 Performance

- o Range: 0.1 to 2000 ppm
- o Detection Limit: 0.1 ppm
- o Sensitivity (max.): 0 to 2 ppm FSD over 100
division meter scale
- o Repeatability: $\pm 1\%$ of FSD
- o Linear Range: 0.1 to 600 ppm
- o Useful Range: 0.1 to 2000 ppm
- o Response Time: less than 3 seconds to reach
90% full scale
- o Ambient humidity: up to 95% relative humidity
- o Operating Temperature: Ambient to 40°C
(instrument is temperature compensated so that
a 20°C change in temperature corresponds to a
change in reading of $\pm 2\%$ full scale at maximum
sensitivity).

3.2 Power Requirements and Operating Times

- o Continuous use on battery: approximately 10
hours
- o Continuous use with HNu recorder reduces
instrument battery operating time to
approximately 5 hours

- o Recharge time: less than 14 hours; a 3 hours charge will charge up to 90% full charge
- o Recharge Current: maximum 0.4 amps at 15 VDC

II. OPERATIONAL PROCEDURE

1.0 Instrument Check-Out

- 1.1 Remove instrument box cover by pulling up on fasteners.
- 1.2 On the instrument panel, there will be a label containing information on light source, calibration date, calibration gas, and span setting.
 - 1.2.1 If the instrument has not been calibrated in the last 14 days or since its last field use, it should be recalibrated. Check the instrument log, which should be maintained with the instrument, for the instrument status and its calibration history. For general use, the instrument should be calibrated to isobutylene at a span setting of 9.8.
 - 1.2.2 Check the label for light source and refer to Table 1 for ionization potentials of various compounds. If the compound you wish to detect is not listed for the light sources provided with instrument, then the light source will have to be changed. Use the probe with the proper light source for the compounds to be detected.
 - 1.2.3 Once it has been determined that the instrument has the correct lamp, the instrument may need to be recalibrated for the specific compound of interest. Use Procedure under 2.1.3 of this Section to calibrate the instrument.
 - 1.2.4 Check the battery supply by connecting the probe to the instrument box, and turning the function switch to the battery check position (Figure 1). (Note: The battery check indicator will not function unless the probe is attached.) The meter needle should deflect to the far right or above the green zone. If the needle is below or just within the green zone or the red LED indicator is on, the battery should be recharged. Follow

the procedure described in Section III (Maintenance and Trouble shooting) to recharge the battery.

1.2.5 Repack the instrument for shipment to the field.

2.0 Field Operation

2.1 Calibration

2.1.1 Equipment and Materials

o Calibration Gas (2 ranges)

Low range 0-20 ppm and mid-range 20-200 ppm of isobutylene gas are used for standard field operation when contaminants are unknown or a mixture of gases is present. The isobutylene gas is used for general calibration because of the instrument's relatively high sensitivity to it and the non-toxic nature of the gas.

Note: A specialty gas may be required if a single atmospheric contaminant is present and the contaminant has a sensitivity different from that of the calibration gas (isobutylene).

o Tubing and fittings (see Figure 1).

o Rotometer or bubble flow meter.

o Field Log, calibration form, and data reporting form.

o Table 1 for ionization potentials for compounds of interest.

2.1.2 Calibration Frequency

This instrument should be calibrated after each field use and prior to each field use. Continuous calibration check should be performed frequently during field operation (for example, check the instrument zero and calibration after every 10 measurements) and document the results properly. Caution: Do Not Change the Settings.

2.1.3 Calibration Procedure

2.1.3.1 Use a three-points procedure to facilitate the proper instrument calibration over appropriate operating ranges. Distinct mixtures of calibration gas with known concentration for selective operating range should be used for calibration. Each mixture should give a 3/4 scale deflection in its respective operating range.

2.1.3.2 Instrument Setup.

- Step 1: Remove Instrument cover by pulling up on the side straps.
- Step 2: Prior to calibration, check the function switch (Figure 2) on the control panel to make sure it is in the OFF position. The probe nozzle is stored inside the instrument cover. Remove cover plate by pulling up on the pins that fasten the cover plate.
- Step 3: Remove the nozzle from the cover. Assemble probe by screwing nozzle into casing.
- Step 4: Attach probe cable to instrument box inserting 12 pin interface connector of the probe cable into the connector on the instrument panel. Match the alignment keys and insert connector. Turn connector in clockwise direction until a distinct snap and lock is felt.
- Step 5: Turn the function switch to the Battery Check position. When the battery is charged, the needle should read within or above the green battery arc on the scale plate. If the needle is below the green arc or the red LED light comes on, the instrument should be recharged prior to making any measurements. Implement steps in Section III to recharge battery.
- Step 6: Turn the function switch to the ON position. In this position, the UV

light source should be on. To verify, gaze at the end of the probe for a purple glow. Do Not Look Directly at the Lamp Itself. If the lamp does not come on refer to Maintenance Step in 2.2 (Section III).

Step 7: To zero the instrument, turn the function switch to the standby position and rotate the zero potentiometer until the meter reads zero. Clockwise rotation of the zero potentiometer produces an upscale deflection while counter clockwise rotation yields a downscale deflection. (Note: No zero gas is needed since this is an electronic zero adjustment.) If the span adjustment is changed during instrument calibration, the zero should be rechecked and adjusted. If necessary, wait 15 to 20 seconds to ensure that the zero reading is stable. Readjust as necessary.

2.1.3.3 Calibration Steps

- Step 1: Insert one end of T tube (Figure 1) into probe. Insert second end of probe into calibration gas in the 20-200 ppm range. The third end of probe should have the rotometer (bubble meter) attached.
- Step 2: Set the function switch in the 0-200 ppm range. Crack the valve on the pressured calibration gas container until a slight flow is indicated on the rotometer. The instrument will draw in the volume required for detection with the rotometer indicating excess flow.
- Step 3: Adjust the span potentiometer so that the instrument is reading the exact value of the calibration gas. (Calibration gas value is labeled on the cylinder).
- Step 4: Turn instrument switch to the standby position and check the electronic zero. Reset zero

potentiometer as necessary
following step 7 of 2.1.3.2.

- Step 5: Record on form and field log all original and readjusted settings as specified in the form.
- Step 6: Next, set the function switch to the 0-20 ppm. Remove the mid-range (20-200 ppm) calibration gas cylinder and attach the low range (0-20 ppm) calibration gas cylinder as described above.
- Step 7: Do not adjust the span potentiometer. The observed reading should be ± 3 ppm of the concentration specified for the low range calibration gas. If this is not the case, recalibrate the mid range scale repeating Step 1 thru 6 above. If the low range reading consistently falls outside the recommended tolerance range, the probe light source window likely needs cleaning. Clean window following Step 2 under 2.3 (Section III). When the observed reading is within the required tolerances, the instrument is fully calibrated.

2.2 Sample Measurement

- Step 1: Place function switch in 0-20 ppm range for field monitoring. This will allow for the most sensitive, quick response in detecting airborne contaminants.
- Step 2: Before entering a contaminated area, determine background concentration. This concentration should be used as a reference to readings made in the contaminated area. Under no circumstance should one attempt to adjust the zero or span adjustments while the instrument is being operated in the field.
- Step 3: Take measurements in contaminated area, recording readings and locations. Should readings exceed the 0-20 scale, switch the function

switch to the 0-200 or 0-2,000 range as appropriate to receive a direct reading. Return the instrument switch to the 0-20 range when readings are reduced to that level. Record measurements in notebook or on an appropriate form.

Step 4: Keep in mind health and safety action guidelines for the level of protection you are wearing. Sustained readings above a certain level may force you to vacate an area or upgrade your level of protection.

Note: The instrument will not function properly in high humidity or when the window to the light housing is dirty. If the instrument response is erratic or lower than expected.

Step 5: When finished, use the reverse Steps 1 thru 5 of Section 2.1.3.2 (Instrument Setup) to shut down the instrument.

III. MAINTENANCE AND TROUBLE-SHOOTING

1.0 Battery Recharging

- 1.1 The instrument should be recharged 1 hour for each hour of use or overnight for a full day's use. (The battery will last 10 hours on a full charge.)
- 1.2 To recharge the battery (or instrument):
 - 1.2.1 Turn the function switch to the off position.
 - 1.2.2 Remove the charger from the instrument top compartment.
 - 1.2.3 Place the charger plug into the jack on the left side of the instrument box.
 - 1.2.4 Connect the charger unit to a 120 V AC supply.
 - 1.2.5 Check charger function by turning the instrument switch to the battery check position. The meter should go upscale if

the charger is working and is correctly inserted into the jack.

1.2.6 Place instrument in instrument mode and charge for the appropriate time period.

1.2.7 Turn the instrument off following the recharge cycle. When disconnecting charger, remove from 120 V AC supply before removing the mini phone plug.

2.0 General Fault Determination and Correction

2.1 Battery level is low. Recharge if necessary implementing steps described under 1.0 (Section III). If the battery will not recharge, it will have to be replaced.

2.2 UV Lamp function - Gaze at sample inlet when mode switch is on an instrument function position and observe for purple glow of lamp. If the lamp does not glow in any of the three instrument function positions, it may be burned out and will have to be replaced. To replace the lamp:

1. Turn the function switch to the off position and disconnect the probe connector from the readout unit.
2. Remove the exhaust screw found near the base of the probe (Figure 3).
3. Grasp the end cap in one hand and the probe shell in the other and gently pull to separate the end cap and lamp housing from the shell.
4. Loosen the screws on the top of the end cap and separate the end cap and ion chamber from the lamp and lamp housing. Care must be taken so that the ion chamber does not fall out of the end cap and the lamp does not slide out of the lamp housing.
5. Turn the end cap over in your hand and tap on the top of it; the ion chamber should fall out of it.
6. Place one hand over the top of the lamp housing and tilt slightly. The light source will slide out of the housing.
7. Replace lamp with one of same energy source as the one removed by sliding it into the

housing. Note: The amplifier board and instrument circuitry are calibrated for one light energy.

8. Place the ion chamber on top of the lamp housing, checking to ensure that the contacts are aligned.
 9. Place the end cap on top of the ion chamber and replace the two screws. The screws should be tightened only enough to seal the "O" ring. Do not overtighten.
 10. Line up the pins on the base of the lamp housing with the pins inside the probe shell. Gently slide the housing assembly into the probe shell. Do not force the assembly as it only fits one way.
 11. Replace and tighten the exhaust screw.
 12. Reconnect the 12 pin connector and turn instrument mode switch to a function position. Check for glow of lamp. If lamp still does not function, the instrument has an electrical short or other problem that will have to be corrected at the factory.
- 2.3 Instrument appears to be functional, but responses are lower than expected or erratic. The window of the light source may be dirty and need to be cleaned. To clean the light source window:
1. Disassemble the probe assembly by repeating Steps 1 thru 6 under 2.2 above.
 2. Clean the window of the light source using compound provided with instrument and soft clean cloth. Important: Use cleaning compound on the window of the 10.2 eV lamp only. The cleaning compound may damage the windows of the 9.5 and 11.7 eV lamps.
 3. Reassemble the probe assembly repeating Step 7 through 12 above.

3.0 Specific Faults

- 3.1 No meter response in any switch position (including BATT CHK)
1. Broken meter movement: Tip instrument rapidly from side to side. Meter needle should move freely, and return to zero.

2. Electrical connection to meter is broken: Check all wires leading to meter and clean the contacts of quick-disconnects.
 3. Battery is completely dead: Disconnect battery and check voltage with a volt-ohm meter.
 4. Check 2 amp fuse.
 5. If none of the above solves the problem, consult the factory.
- 3.2 Meter responds in BATT CHK position, but reads zero or near zero for all others.
1. Power supply defective: Check power supply voltages per Figure 4. If any voltage is out of specification, consult the factory.
 2. Input transistor or amplifier has failed: Rotate zero control; meter should deflect up/down as control is turned. Open probe; both transistors should be fully seated in sockets.
 3. Input signal connection broken in probe or readout: Check input connector on printed circuit board. Should be firmly pressed down. Check components on back side of printed circuit board. All connections should be solid, and no wires should touch any other object. Check all wires in readout for solid connections.
- 3.3 Instrument responds correctly in BATT CHK, and STBY, but not in measuring mode.
1. Check to see the light source is on (See Section 2.2).
 2. Check high voltage power supply (See Figure 4).
 3. Open end of probe, remove lamp and check high voltage on lamp contact ring.
 4. If high voltage is present at all above points, light source has most likely failed. Consult the factory.
- 3.4 Instrument responds correctly in all positions, but signal is lower than expected.

1. Check span setting for correct value.
 2. Clean window of light source (See 2.3).
 3. Double check preparation of standards.
 4. Check power supply 180 V output. See Figure 4.
 5. Check for proper fan operation. Check fan voltage. See Figure 4.
 6. Rotate span setting. Response should change if span pot is working properly.
- 3.5 Instrument responds in all switch positions, but is noisy (erratic meter movement).
1. Open circuit in feedback circuit. Consult the factory.
 2. Open circuit in cable shield or probe shield. Consult the factory.
- 3.6 Instrument response is slow and/or irreproducible.
1. Fan operating improperly. Check fan voltage. See Figure 4.
 2. Check calibration and operation.
- 3.7 Low battery indicator.
1. Indicator comes on if battery charge is low.
 2. Indicator also comes on if ionization voltage is too high.

APPENDIX G-8

CALIBRATION AND MAINTENANCE OF THE HCN MONITOX DETECTOR

Bedienungsanleitung Operating Instructions

COMPUR 4100 SD Monitox HCN

MDA Scientific, Inc.
405 Barclay Blvd.
Lincolnshire, Illinois 60069
Phone: 800-323-2000 (in IL 312/634-2800)
Telex: 72-6399 MDA-USA • Fax: 312-634-1371

Compur-Electronic GmbH

5.6.

Hinweise zur Fehlersuche

Fehler	Hinweis
Batterie/Delektor geht nicht	Batterien wechseln (5.1.)
Funktionstest mit Generator geht nicht	a) Evtl. mit 2. Detektor nachprüfen, ob Generator o. k., sonst b) b) Filterkappe wechseln (5.2.), wenn nicht verschmutzt, c) c) neue Sensorzelle einbauen (5.3.).
Generator liefert nicht genug Gas	Zelle beleuchten, d. h. mit Fauchthallekappe mehrere Tage stehen lassen, sonst Generatorzelle wechseln (5.4.).
Am Generator leuchtet rote LED beim Test auf	Generatorbatterie ersetzen (5.5.).

6.

Zubehör und Verbrauchsmaterial

Bestellnummern für Verkaufseinheiten

1. Gasdetektor HCN Digitalanzeige, 2 Alarmschwellen mit Dosimeter-Anschluß	U 5306 203
2. Zelle HCN mit Filterkappe	U 5800 103
3. Filterkappe HCN (10 Stück)	U 5810 341
4. Batterie PX 23 (1 Stück)	U 4990 001
5. Gasgenerator HCN	U 5390 300
6. Generatorzelle HCN	U 5820 300
7. Kalibrierkassadapler	U 5900 106
8. Meßleitung: Eichen	U 5900 112
9. Digitalvoltmeter	U 5900 018
10. Stromgenerator	U 5900 023
11. Kalibrierkabelsatz für Stromgenerator	U 5900 125
12. Protokollheft	U 5900 004
13. Ohrhörer	U 5900 002

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Gas Detection and Warning System COMPUR 4100 SD Monitox HCN

- The COMPUR 4100 SD Monitox Gas Detection and Warning System comprises
- 1 gas detector (alarm unit with digital display)
 - 2 gas generator (test unit)
 - 3 console (recommended accessory)
 - 4 kit of tools (recommended accessory)

The system is especially designed to monitor the TLV of HCN.

CAUTION!

Although the 4100 SD Monitox for HCN has been highly simplified for ease of operation by the user, it is nevertheless a complex measuring instrument which will operate reliably only if these operating instructions are carefully observed and if the instrument is checked regularly by the safety officer.

This applies in particular to the regular replacement of the cells and daily functional tests. The responsibility for any changes made in the alarm threshold settings must be borne entirely by the operator; COMPUR recommends the strict observance of the TLV. Since the unit is designed to be intrinsically safe, all repairs must be made by the manufacturer or other approved personnel.

COMPUR offers the instrument with the following factory settings:

- first alarm threshold = at TLV = 10 ppm
- second alarm threshold = at 2 TLV = 20 ppm

The detector cell will be destroyed if the detector is permanently exposed to a HCN-concentration exceeding 1000 ppm. In this case the cell has to be replaced.

1.

Technical Description of the COMPUR 4100 SD Monitox Detector for HCN

1.1.

Applications

The COMPUR 4100 SD Monitox is a personal monitor for HCN.

It is designed to be worn attached to the clothing near the breathing zone of the person to be protected. The detector produces an audible first alarm when the HCN-concentration exceeds the TLV (factory setting: 10 ppm) and a second alarm, when it exceeds 2 x TLV.

Independent of the alarm setting, the digital display shows the actual HCN-concentration in ppm (parts per million) in the nominal range of 0 - 100 ppm HCN.

In conjunction with the COMPUR 4102 Dosimeter, the unit can be employed to register HCN-concentrations at confined spaces ranging from 0 to 10 x TLV.

The COMPUR 4100 SD Monitox cannot be used to measure process gas streams or in presence of continuous high HCN-concentrations.

1.2.

Mode of Operation

Ambient air diffuses through the filter insert (a dust filter) (5) to the measuring cell. The measuring cell, a dual-electrode electrochemical cell with an organic electrolyte gel, generates an output current proportional to the partial pressure of HCN in the air.

A series of electronic amplifiers supply a voltage signal which is fed to the comparator for the alarm threshold. If the first alarm threshold is exceeded, an intermittent tone is produced; if the second alarm threshold has been exceeded a dual-tone signal is produced by the tone generator and loudspeaker (or earphone in very loud areas). The standardised analog signal corresponding to the actual HCN-concentrations (the TLV corresponds to 80 mV) can be fed to the Dosimeter.

The same signal is fed to the AD-converter driving the digital display. The display is adjusted to give a reading of 10 ppm at 80 mV input.

The 4100 SD Monitox consists of two separate power circuits (via two miniature batteries); the circuit for the analogue part is separated from that for the alarm-generation.

When the „on-off“ switch is moved to the „Batt.“ position, the batteries will be tested before the instrument is turned on. In this switch position, the batteries are electronically tested under the high load of the final tone stages. If one of the batteries fails to reach the predetermined lower theoretical limit, no alarm will be heard.

1.3.

Technical Data for the COMPUR 4100 SD Monitox for HCN

Conformity certificate	BVS 82.013
Safety class	EEEx Ib II CT 6
Dimensions	104,4 x 82 x 24 mm
Weight (with batteries)	approx. 150 g
Power supply	2 x PX 23 (5,6 V)
Battery service life	approx. 1000 h.
Display range	0 - 100 ppm
Alarm volume	min. 80 dBA / 30 cm
Alarm levels	2 alarms, adjustable
Response time	$T_{90} < 10$ s $T_{95} < 3$ min.
Time to alarm	20 ppm < 15 s 50 ppm < 3 s
Connection possibilities	earphone, dosimeter
Temperature range	0 - 50 °C
Relative humidity	10% - 95%
Zero point drift	< 1 ppm / 6 months
Sensitivity drift	$< 15\%$ / 6 months
Service life of the cell	min. 6 months (dependant on dose)

1.4.

Cross-sensitivities

Test components	Test concentration	Indication in ppm HCN
SO ₂	5000 ppm / 40% RH	10
NO ₂	10 ppm	- 6
NH ₃	1000 ppm	10
CO	1000 ppm	—
CO ₂	1000 ppm	—
H ₂	1000 ppm	—
CH ₂ :CHCN	10 ppm	—
CH ₃ CN	200 ppm	—
(CH ₃) ₂ N	500 ppm	17
CH ₃ OH	200 ppm	—
COCl ₂	5 ppm	10
Cl ₂	10 ppm	5
HCl	10 ppm	7
H ₂ S	2 ppm	10
Hydrocarbons, saturated	2% vol.	—
Hydrocarbons, unsaturated	1% vol.	—
Aromatic compounds (also alkylated)	200 ppm	—

2.

Technical Description of the COMPUR 4100 SD Monitox Gas Generator for HCN

2.1.

Applications

The HCN gas generator serves to enhance the reliability of the Monitox gas detection and warning system. The Monitox detector must undergo a functional test by placing it on the generator before each use. The generation of a gas concentration exceeding the TLV ensures that the detector will respond reliably during use (picture 2).

The gas generator, however, is not designed to generate a calibration gas of known concentration. Daily testing of the Monitox detector does not mean that the user is not recommended to change cell sequentially.

The COMPUR 4100 Gas Generator must not be exposed to or used in explosive atmospheres.

N.B.: The generator cell may dry out at very low relative humidity in the air. In this case, it is necessary to put the moisture cap delivered with the generator on top of the generator always when it is not in use.

This ensures a correct gas concentration for the detector test.

2.2.

Mode of Operation

The switch on the generator is activated by placing the detector in the matching recess on the generator head.

A small fan feeds a flow of air past the generator cell directly to the detector cell. At the same time, gas is generated electrolytically in the generator cell in such an amount that the gas concentration is high enough to cause the detector to respond within 10 seconds (alarm threshold 10 ppm). The period of gas generation is indicated by the green LED.

The red LED indicates when the battery must be replaced.

After a 10 seconds interval, gas production is terminated and the fan conveys pure air until the detector is removed.

This functional test of the detector checks any of the following defects:
clogging of the dust filter
a malfunctioning cell
a malfunctioning electronics system
a malfunctioning generator.

2.3.

Technical Data of the COMPUR 4100 SD Monitox Gas Generator for HCN

Dimensions	133 x 65 x 40 mm
Weight (incl. batteries)	approx. 250 g
Temperature range	0 °C – 50 °C
Power supply	9 volt alkali battery, leakproof, e.g. Mallory 1604
Generator cell service life	approx. 3000 tests or for 1 year
Battery service life	approx. 3000 tests

3.

Use of the detector and generator

3.1.

Detector actuation and functional test

Battery Test

Turn the switch on the COMPUR 4100 SD Monitox to "Batt.". If the battery has sufficient power to operate the detector for eight hours, an audible (intermittent) tone will be heard. The LCD-display is switched off at the "Batt." test position. If no tone is emitted, this indicates that at least one of the batteries is exhausted. For safety reasons both batteries should be replaced (refer to section 5.1.).

When the audible tone has been heard (to preserve batteries, the test should be as short as possible), the switch is moved to "ON". The tone will cease. The LCD-display is operating now. It must show "0" ppm after some seconds.

Functional Test (picture 2)

Place the detector on top of the generator as illustrated.

As soon as the detector sounds its alarm, it must be removed from the generator. The detector is ready for operation once the alarm has ceased.

If the detector alarm does not sound within ten seconds the detector has to be checked and serviced. If necessary, the filter cap has to be replaced (see point 5.2.).

It is advisable to record the test and assignment of the gas detector in the detectolog.

The battery test and functional test must be performed prior to each use to thus ensure maximum safety.

During the gas test the LCD-display must show the response of the cell to HCN-concentration as well. As the alarm threshold is factoryset at 10 ppm the alarm should sound at 10 ppm. As the display reads a new value every second, the time for alarm and display of 10 ppm may be different.

1.2.

Use of the Gas Detector

The gas detector must be worn in the breathing zone of the person to be protected and the filter cap (5) should not be covered any way.

The rubber lip on the carrying clip makes it possible to securely attach the Monitox to articles of clothing (e.g. the breast pocket).

This is not deemed adequately secure, the device supplied with the Monitox can be secured in the holes of the carrying clip. This enables the Monitox to be worn around the neck.

At all possible, the filter cap should be protected from water, dust-laden air or dirt. With the battery and functional tests (refer point 3.1.) should be performed before the detector is put into operation.

HCN gas concentration in the vicinity of a sensor exceeds the set alarm value, the alarm will sound after a delay dependent on the gas concentration (The higher the concentration, the more quickly the COMPUR 4100 SD Monitox will respond).

The alarm sounds at a level of at least 80 dB at a distance of about 30 centimeters (12 inches).

3.3.

Connecting the Earphone

When the detector is being utilised in an area with high background noise, the optional earphone should be used to be sure that the alarm will not go unnoticed. The earphone is connected to the earphone socket (8) on the detector. This socket disconnects the internal loudspeaker. If the earphone is being used, it is important that the tests also be conducted with the earphone plugged into the detector (refer to point 3.1.). When the earphone is not being used, the socket should be closed with the plastic plug.

3.4.

Connecting the Dosimeter

The COMPUR 4102 Mini-Dosimeter can be connected to the 4100 SD (refer to operating instructions for the 4102).

The generator test can also be carried out with the Dosimeter connected to the COMPUR 4100 SD if the detector is turned 180° about its longitudinal axis relative to the position shown in point 3.1. and then placed on the generator in that way, that the cell fits into the recess on the generator. The functional test is then started by pushing the generator button with one's finger.

The plug should be replaced in the Dosimeter socket whenever the Dosimeter is not being used.

3.5.

Digital Display

In addition to the warning-function of the COMPUR 4100 SD Monitox its digital display (6) gives a direct reading of the actual HCN concentration.

Thus it is possible to determine HCN-concentrations below and above the TLV-level, giving the skilled worker and industrial hygienist the means to detect unusual conditions of HCN-concentrations with high accuracy and resolution.

The COMPUR 4100 SD Monitox is, however, even with its digital display, primarily a measuring and warning device for personal protection.

It has not been designed for measurement in process-control; moreover exposures to high HCN-concentrations for any length of time must be avoided, as the accuracy of the reading will suffer.

3.6.

Detector Deactivation and Storage

- a) brief period of inactivity (up to a month) the detector is deactivated (switch to "OFF")
- b) Prolonged inactivity and storage
It is advisable to open the Monitox and remove both the cell and batteries, to provide them from leaking and corroding the interior of the Monitox (refer to the sections on cell and battery replacement 5.3.).
Before reutilizing the Monitox a new cell has to be installed.

4.

Calibration Instruction for the detector COMPUR 4100 SD Monitox

To enhance the intrinsic accuracy of the detector for HCN it is necessary to calibrate the detector either with a HCN nitrogen mixture with definite concentration of HCN or make an electronic adjustment by means of the COMPUR current generator U 5900 023.

4.1.

Accessories required

1.1.1.

Calibration with gas

- a) calibration cap to place onto Monitox
- b) flow meter
- c) millivoltmeter 0 - 2000 mV; input resistance $\geq 1 \text{ M } \Omega$
- d) tubing, set of test cables, screw-driver
- e) calibration gas, known concentration, about 10 ppm HCN in pure N_2

Itemark: The generation and above all the stability time of HCN calibration gas is not without problems. So if only a small number of detectors are to be calibrated, the electronic method should be preferred.

1.1.2.

Calibration - Electronical Method (picture 6 and 7)

- a) calibration unit (current generator)
- b) millivoltmeter 0 - 2000 mV, input resistance $\geq 1 \text{ M } \Omega$
- c) set of test cables, screw-driver

4.2.

Zero calibration and gain adjustment with calibration gas

4.2.1.

Preparation

The Monitox is opened and positioned with the electronic components upward on a non-slip surface. The cover with the digital display is carefully put aside with the display upward.

Then the unit is switched on via "Batt." position to "ON". The LCD-display should read 00 ppm after several seconds.

The excellent zero-point stability of the cells will normally make unnecessary to adjust the zero-point. Deviations from zero are caused mostly by fault sensor cells.

For zero-checking remove sensor cell.

4.2.2.

Zero-Adjustment

Connect Millivoltmeter to tie down point (MP 2) and GND (MP 1) (picture 6). If the reading is not zero in clean air, and also is not zero without sensor cell, potentiometer (R 9) (offset voltage) has to be varied until the reading is zero.

Note: If reading is zero without cell and not zero with the cell, it may need up to one hour to stabilize the cell. If a cell has been removed for a longer period without short-circuiting the two connectors, the time to stabilize may be up to one day. A new cell therefore has short-circuit on the small pcb, that must be broken away before inserting the cell.

4.2.3.

Gain Adjustment with gas

The special calibration adapter is tightly put onto the dust filter on top of the detector cell.

Adjust a calibration-gas flow through the calibration cap; flow rate should be approx. 100 cm³ per minute and the inlet must be the smaller pipe; to avoid pressure variations the outlet should be free of obstacles. After 5 minutes the display of the Monitox has reached its final value.

Connect millivoltmeter to tie down point (MP 2) and GND (MP 1). Depending on the concentration of the calibration gas the following voltage should be displayed: (adjust by means of pot R 7)

$$U = \frac{[c]}{10 \text{ ppm}} \times 80 \text{ mV}$$

The display of the Monitox must show the gas concentration. In the opposite, adjust pot (R 15) until correct reading is shown.

4.2.4.

Gain-Adjustment with the current calibrator

Each detector cell produced by COMPUR is supplied with an indication of the output current at 10 ppm HCN. (Never throw away packings of replacement cells before having noted this indication!!!)

Remove detector cell. Insert calibration cable with the plug board into plug connector for detector cell. The gold contacts must touch the spring contacts. Connect other side of the cable to the current generator.

Make sure of correct polarity of plugs. Switch on generator, turn button till generator display shows output current of detector cell.

Remark: Display always shows actual value of current. If it is zero, check the contacts!

Connect voltmeter to tie down point (MP 2) and GND. Adjust sensibility by means of pot (R 7) until 80 mV is displayed. Monitox must now display 10 ppm. In opposite, adjust pot (R 15).

4.3.

Setting the Alarm thresholds

The alarms of the standard version are to be set on 10 ppm (first alarm 1 TLV) and 20 ppm (second alarm 2 x TLV).

To set the alarm levels, push the 2 mini-switches (S 1) to the right. The display of the Monitox shows now the level of the 1st alarm threshold. This can be adjusted by means of the potentiometer (R 30).

To adjust the 2nd alarm level, push the upper switch to the left. The display shows now the 2nd alarm threshold. This can be adjusted by means of the potentiometer (R 29).

After having adjusted the alarm levels, push both mini switches to the left. The Monitox display shows now the actual concentration of HCN.

4.4.

Concluding the adjustment operations

After the settings have been made, turn the switch on the pcb to "OFF"-position. Make sure that the switch-handle on the cover is also in the "OFF"-position. Then carefully replace the cover and fold the connecting cable between pcb and display so that it is neither squeezed in nor cracked. Tighten the screws. The Monitox is now ready for operation.

5. Maintenance and Servicing Instructions

5.1.

Battery Replacement

1. Turn switch (7) to "OFF".
2. Remove three screws (12).
3. Turn detector over and remove front cover.
Attention: Do not attempt to remove the cable between front panel and pcb!
4. Lift out battery housing, disconnect plug.
5. Unscrew and remove battery lids.
Replace batteries with + pole towards lid.
Replace lids.
6. Plug-in battery plug. Ensure cable and cable socket in right position.
7. Replace battery housing and front cover, carefully adjust the cable of the front panel, so that it is not damaged by fixing the front panel; then tighten the screws.
8. Repeat battery test.

5.2.

Filter Cap Replacement

1. Remove screws (7) (picture 4) and open detector.
2. Carefully remove sensor cell together with filter cap (5). Pull cap off cell (4).
3. Attach new filter cap (with identical gas label HCN) and return sensor cell to original position.
Filter cap order number appears on plate attached to inside of front panel and is listed in section 6.
4. Replace front cover and tighten screws
5. Repeat performance test.

5.3.

Sensor Cell Replacement

1. Open detector (see 5.1.).
2. Remove cell together with filter cap.
3. Remove new cell and filter cap from storage container, pull transparent cap off the cell and replace this by the new filter cap. Correct position of filter cap is shown in illustration.
Remove short-circuit protection attached to pcb by breaking it away.
4. Proceed current calibration (4.2.4.).
5. Replace sensor cell with filter cap in proper position.
6. Close Monitor.

5.4.

Generator Cell Replacement (picture 5)

1. Open housing (as when replacing battery)
2. Unsolder fan leads (10).
3. Loosen four screws (11) and three screws (12).
4. Remove outlet, gas cell and fan through the front.
5. Carefully insert replacement unit U 5820 300 consisting of outlet, cell and fan and tighten screws (12).
6. Tighten screws (11). Align circuit board so that pin (13) reliably actuates switch (14) when gas detector attached.
7. Resolder fan leads (10).
8. Reassemble generator and tighten screws.
9. Testing: Use properly functioning gas detector for same gas. Switch to "ON", attach. Alarm must sound after about eight seconds.

5.5.

Generator Battery Replacement

Loosen four screws on rear housing panel. Carefully remove front cover. For correct positioning of battery, refer to illustration 5.

5.6.

Troubleshooting

Malfunction	Remedy
Battery test: no response	Replace batteries (5.1.)
Generator test: no response	a) Repeat test using 2nd detector, if no response, b) Replace filter cap (5.2.), if not dirty, c) Insert new sensor cell (5.3.)
Generator does not supply enough gas	Use moisturizing cap for several days, otherwise replace generator cell (5.4.).
Red LED lights up during test	Replace generator batteries (5.5.).

6.

Accessories and consumables

Part Numbers

1. Gas detector digital display, 2 alarm thresholds with Dosimeter output	U 5306 203
2. HCN cell with filter cap	U 5800 103
3. HCN filter cap (10 pcs.)	U 5810 341
4. Battery PX 23 (1 pc.)	U 4990 001
5. HCN gas generator	U 5390 300
6. HCN generator cell	U 5820 300
7. Calibration gas adapter	U 5900 106
8. Measuring cable: calibration	U 5900 112
9. Digital Voltmeter	U 5900 018
10. Current calibrator	U 5900 023
11. Calibration cable used in connection with current calibrator	U 5900 125
12. Detectolug	U 5900 004
13. Earphone	U 5900 002

APPENDIX G-9

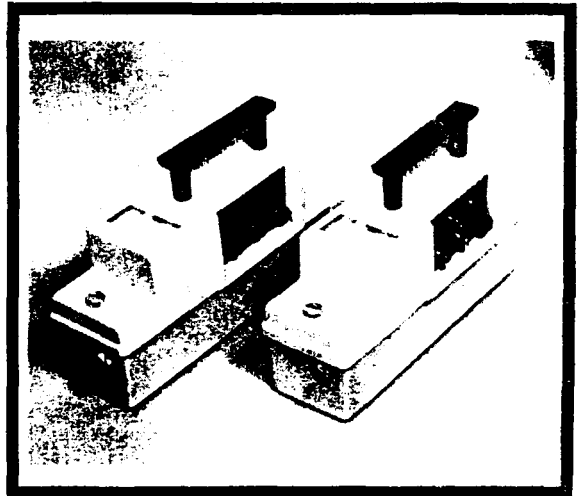
CALIBRATION AND MAINTENANCE OF THE GAS-TECH DETECTOR

MODEL 1939OX

GAS|TECH

MODELS 1238 & 1314
GAS|TECHTOR

INSTRUCTION MANUAL



MADE BY

GASTECH INC

8445 CENTRAL AVE., NEWARK, CA 94560

WARNING

Explosive gas mixtures can maim, disfigure and kill, toxic vapors can cause impairment of health, and oxygen deficient atmospheres can cause instant death. It is essential that users of this instrument read, understand and follow the instructions for operation and maintenance, and the precautions contained in this manual to ensure that the instrument will warn of explosive or oxygen deficient atmospheres.

This instrument is designed and intended to warn personnel of the hazards of combustible gas or oxygen deficiency prior to entry or while occupying spaces subject to such hazards. It can safely be used in testing any mixture of combustible gas in air. The electric circuitry is non-incendive to any gas mixture in air and is certified by the manufacturer to be intrinsically safe against initiation of explosion in Class I Division 1 Group C and D environments.

Explosive mixtures of acetylene or hydrogen in air (Group A and B atmospheres) are unsuitable environments for personnel and for electrically operated instruments. The GasTechtor was not designed for use under such conditions and as such is not certified intrinsically safe for Group A and B atmospheres.

Likewise, use of the instrument with oxygen-enriched mixtures is beyond the normal scope of its intended application.

DO NOT SAMPLE OR TEST OXYGEN-ACETYLENE MIXTURES AS FOUND IN OXY-ACETYLENE WELDING AND CUTTING EQUIPMENT.

DO NOT USE FOR DETECTION OF TOXIC GASES OTHER THAN ORGANIC VAPORS IN THE TOXIC RANGE.

WARNING

INSTRUCTION MANUAL
GASTECHTOR HYDROCARBON SURVEYOR
MODEL 1939-OX PERCENT GAS AND OXYGEN INDICATOR
SERIAL NO. 88910
CALIBRATED FOR METHANE

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- VI. MAINTENANCE
- VII. CIRCUIT DESCRIPTION
- VIII. PRECAUTIONS AND NOTES ON OPERATION
- IX. PARTS LIST
- X. ILLUSTRATIONS

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SPECIAL INSTRUCTIONS

Model 1939 OX Triple Range GasTector
For 0-100% LEL/0-100% by Volume of Methane & 0-25% Oxygen

1939-091786-S1

This instrument is a special version of the Model 1314 triple-range Hydrocarbon Surveyor as described in the accompanying manual pages. However, in place of the ppm range, a second range of 0-100% Methane by volume is provided. Refer to the accompanying manual for general information on the GasTector Model 1314, and refer to these supplementary pages for specific information relating to the Model 1939 OX version.

A. LEL and Oxygen Ranges

The LEL and Oxygen ranges are unchanged from the standard Model 1314, so the accompanying instructions apply.

B. 100% Gas Range

This range is obtained from a set of thermal conductivity filaments, which respond to the cooling effect of methane relative to air, and hence can be used over the full range of concentrations from 0 to 100%. The thermal conductivity section is activated by pressing the range switch in, to the GAS position. This simultaneously turns off the % LEL section.

The thermal conductivity Wheatstone bridge circuit is independent of the LEL circuit, with its own zero adjust and span controls. The zero adjust potentiometer is accessible only while the instrument is opened; the control, marked GAS ZERO, is a slotted-shaft miniature potentiometer found at the front of circuit board, close to terminal strip. It should require adjustment only rarely, and should be set only after circuit has warmed up for 10 minutes or more. (Zero on % GAS scale is however affected by turning the external (% LEL) ZERO potentiometer, so this knob position should not be changed while in the % GAS range.)

Calibration and alarm settings are adjustable, using the potentiometers marked PPM SPAN and PPM ALARM respectively. Span can be set to give a reading of 100% while undiluted propane is admitted to instrument. Alarm can be set as described on Page 13.

Thermal conductivity filaments are mounted in a metal block installed at the outlet of pump, so that total flow passes through it. A change in flow due to restriction of inlet tube will cause a zero shift which must be corrected for best accuracy. Therefore always adjust zero with the hose probe, and any other sampling components connected.

Thermal conductivity filaments connect to a set of terminals found at the end of the sensor block. To replace thermal sensors:

- 1) Disconnect wires at terminals noting wire color and location.
- 2) Remove retaining screws at each corner of the sensor plate.
- 3) Pull out sensor plate with sensors.
- 4) Install new sensor assembly in same position. Be sure gasket is in place.
- 5) Tighten screws snugly.
- 6) Reconnect wires to terminals as noted in 1) above. Turn on, and adjust zero and span.

Filaments will rarely if ever require replacement, as they operate at low temperature.

C. Sample System

- 1) Probe, consists of a 10" long 1/4" OD plastic tube with a dust filter chamber at the upper end, forming a handle. This filter chamber is of transparent acrylic plastic, so the filter condition can readily be inspected. To replace filter, unscrew filter chamber where it connects to the nickel-plated threaded base. Cotton may be used for filter material; pack cotton in firmly but not tight enough to restrict flow. Replace cotton when it becomes wet or dirty.
- 2) Hose, is attached by means of a knurled and threaded fitting on each end.

D. Replacement Parts

The only significant additions to the parts list in the accompanying Instruction Manual are:

<u>Stock No.</u>	<u>Description</u>
17-0491	Adapter, inlet
35-0500	Fitting, inlet
62-0106M	Filaments, thermal conductivity
80-0001	Hose, teflon-lined
80-0150	Probe, 10" plastic

When ordering any spares or replacement parts, always specify Model 1939 and serial number.

INSTRUCTION MANUAL
 GASTECHTOR
 HYDROCARBON SUPER SURVEYOR
 MODEL 1314
 PPM/LEL GAS INDICATOR WITH OXYGEN SECTION
 AND MODEL 1238
 PPM/LEL GAS INDICATOR

Serial No. : _____
 Calibrated for : _____
 LEL Alarm : _____ % LEL
 PPM Alarm : _____ ppm
 Oxygen Alarm : _____ % O₂ (Falling)
 _____ % O₂ (Rising)

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I. GENERAL DESCRIPTION

The Model 1314 GasTechtor Hydrocarbon Surveyor is a battery-powered portable instrument that can detect and indicate concentrations of combustible gas or vapor in air, in the explosibility and parts per million (PPM) ranges, simultaneously with measurement of oxygen and detection of oxygen deficiency. It can detect and indicate gas concentrations up to the lower explosive limit (LEL) and actuate a characteristic audible signal if concentration exceeds a preset level. It also analyzes for oxygen over a range of 0 to 25%, and actuates a different audible signal if oxygen concentration drops below a preset level. In later models, a third characteristic audible signal is actuated if for any reason the oxygen concentration exceeds 25%. Samples of the atmosphere under test are drawn continuously by means of a built-in pump and analyzed for combustible gas on a heated catalytic platinum element. A solid-state amplifier is used to amplify indications of the catalytic element to give adequate meter deflection even on trace gas concentrations. At the same time, the sample passes over an oxygen detector which gives an output in terms of percent oxygen.

Power for the instrument is provided by a built-in rechargeable battery. An extension hose permits withdrawal of samples from specific points or enclosed spaces. An audible alarm, active in both combustibles ranges and in the oxygen range, sounds whenever gas concentration reaches a preset level, and an audible signal is also given in case of malfunction or a dead battery. Instrument is designed to withstand severe service including exposure to water, dust, bumps and rough handling.

The Model 1238 is an equivalent instrument for measurement of combustible gas in two ranges, but without inclusion of the oxygen section. If using the Model 1238, disregard all references to oxygen measurement in this manual.

Primary application is in determining concentrations of various organic vapors around industrial operations. The Surveyor is adaptable for any measurements where small concentrations of combustible gas are to be detected. Other uses are in determining explosion hazard in tanks and other confined spaces, checking for presence of flammable vapors, and investigating cases of suspected arson. With the Model 1314, every test for combustibles or toxic hydrocarbon vapor is automatically accompanied by a test for oxygen deficiency.

II. DETAILED DESCRIPTION

A. Housing

The Model 1314 is housed in a fiberglass case which is durable, shock-resistant, and protected against entry of water. The lower half, containing the batteries, sensors and sample-drawing system, has no openings near the bottom and hence can safely be placed in mud or water up to 4 cm depth without hazard to the internal components.

The upper half contains all of the electronic circuitry, and is provided with a substantial carrying handle. The lip of the upper case overlaps the lower, to shed water. Upper half is clamped to lower by means of a heavy-duty knurled thumbcrew.

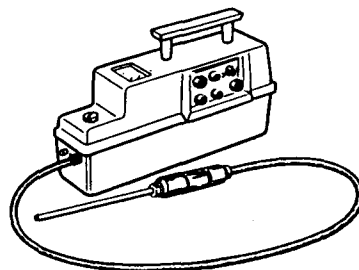


FIGURE 1. MODEL 1314 HYDROCARBON SUPER SURVEYOR

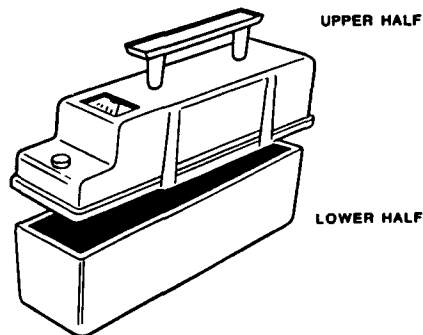


FIGURE 2. MODEL 1314 HOUSING

B. Combustible Gas Sensor

The combustible gas sensitive detector is a replaceable assembly which is installed within an anodized aluminum enclosure or reaction chamber at front of instrument. Detector is retained in chamber by means of a threaded ring, and sealed by an O-ring gasket. Sample enters chamber from the front, flows over the detector, then to the pump. The pump exhaust flows through the oxygen detector chamber and then discharges to the outside through a vent hole in the front.

The active detector element is of the platinum catalyst type, and an electrically identical but non-catalytic reference element mounted in the same environment serves to stabilize the measurement and compensate for effects of non-combustible gases, temperature variations, etc. Elements are protected by a sintered stainless steel flame arrestor, which permits access of the surrounding atmosphere by diffusion, but prevents outward propagation of flame should an explosive atmosphere be sampled. Flame arrestor also acts as a diffuser to isolate elements from flow fluctuations.

Detector assembly connects electrically to circuit board by means of three screw terminals, accessible when upper half of housing is removed.

C. Oxygen Sensor

The oxygen-sensing detector is an electrochemical cell in which gold and lead electrodes are immersed in an alkaline electrolyte, and covered by a permeable fluorocarbon membrane. Oxygen from the surrounding atmosphere diffuses through the membrane and enters into an electrochemical reaction whose rate is directly proportional to the partial pressure of oxygen, the end product of this reaction being lead oxide. The current generated by this reaction is amplified and used to drive the meter and the alarm circuit.

The detector is clamped into a cavity in an anodized aluminum block, through which the sample flows after it leaves the pump.

D. Meter

Indications of the instrument are displayed on a meter, visible through a window on top face of instrument case. On LEL, the insensitive combustibles scale, meter reads gas concentration in units of explosibility, 0-100% LEL, where 100% LEL represents the minimum concentration of gas in air, below which propagation of flame does not occur when placed in contact with an ignition source.

A mark on scale, "BATT CK", represents the minimum permissible battery voltage, as an indication of state of charge of the battery.

A second scale, 0-500 ppm, is provided for use when instrument is in the PPM range. (0-1000 ppm is an optional range.)

A third scale, for oxygen, reads 0-25% oxygen. A mark on scale, "O₂ CAL", corresponds to the normal oxygen content of atmospheric air, 21%.

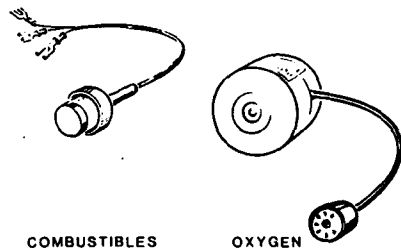


FIGURE 3. SENSORS

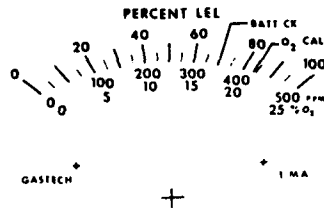


FIGURE 4. METER DIAL

E. Controls & Indicators

1. The six controls that are used in normal operation of the instrument are arranged on the left side of instrument as viewed from the rear. These controls are recessed to minimize possibility of accidental operation.

- a) POWER switch, an alternate-action push button switch which energizes circuit when pressed. An orange indicator dot is exposed when the switch is in the ON position, serving as a mechanical pilot light.
- b) BATTERY CHECK, a momentary push button switch, when pressed connects meter as a voltmeter for battery condition check.
- c) PPM/LEL ZERO, a potentiometer which is used to adjust combustibles circuit to read zero in the absence of combustible gas. The potentiometer shaft may be fitted with a control knob for convenience in adjustment, or optionally with a screwdriver slot to minimize accidental change.
- d) LEL-PPM switch, an alternate-action push button used to select combustibles operating range. When out, the instrument is in LEL range. When in, as indicated by the colored indicator dot, circuit is in the sensitive ppm range.
- e) OXY-PPM/LEL switch, an alternate-action push button switch which selects the operating range, either combustibles (PPM/LEL) in the "out" position or oxygen (OXY) in the "in" position. A colored indicator dot shows when the switch is "in". (Not found on Model 1238)
- f) OXY CAL, a potentiometer which is used to adjust circuit to read 21% when detector is surrounded by known normal air. It also may be fitted with a knob or an optional slotted shaft. (Not found on Model 1238)
- g) Other controls, used in adjustment but not in routine operation, are described in Circuit Board Section below.

2. Indicators

Alarm lights, red and amber, illuminate when the corresponding section of instrument is in alarm condition. Red light blinks in an on-off pattern of equal length pulses, to show that instrument is in combustible gas (LEL or PPM) alarm condition. Amber light blinks in a short-long pattern to show that instrument is in abnormal oxygen condition. (Not found on Model 1238)

F. Buzzer

A solid-state electronic buzzer is mounted at the rear of instrument, behind perforations which permit transmission of sound. The buzzer gives a pulsed tone on detection of gas or oxygen deficiency, and a continuous tone in case of excess oxygen or a malfunction, either low battery voltage or downscale drift of meter. The audible tone pulses correspond to the visual pulses of the corresponding alarm light, except in the case of excess oxygen which gives a steady tone.

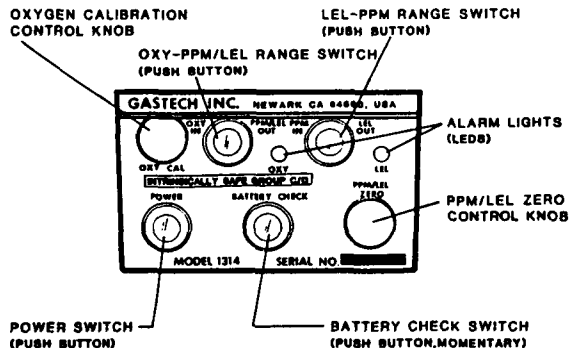


FIGURE 5. CONTROLS AND INDICATORS

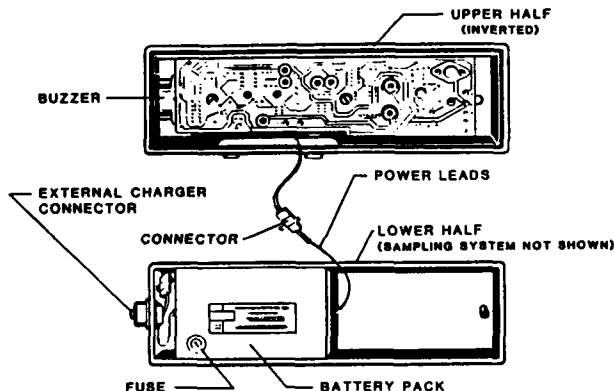


FIGURE 6. INTERIOR OF UPPER AND LOWER HALF

C. Batteries

The battery pack, consisting of seven 3.5 ampere-hour nickel-cadmium cells in series, is secured within lower half of case. The cells are sealed as a unit, either with threaded bushings in bottom for clamping to instrument case, or with holes all the way through to accommodate 3"-long screws and a hold-down bar. Power output (red and black) leads extend from front end of pack, and terminate in a plastic plug connector which mates with a connector on the main circuit board. A similar connector at rear connects to the charger socket, so that battery may be unplugged at both ends for convenient removal. Current limiting resistors sealed into the pack limit maximum current that can be drawn on short circuit. Battery pack will power the instrument for approximately 10 hours. A protective fuse (type 3AG 1A) is installed, in a recessed fuseholder set into top surface of pack, and serves as an added protection against short circuit or overload.

GasTector instruments may be supplied with the 49-8051 Battery Pack with replaceable cells. This design was developed for the convenience of replacing any cell that may fail, in lieu of having to replace an entire battery pack. This battery pack is permanently secured by two screws through the bottom of the case. Instruments bearing the UL classification label always use this pack.

Battery packs manufactured 1987 and later may have a third (orange) wire connected to the output plug. It has no function in Model 1238/1314.

H. Circuit Board

All circuit components are arranged on two epoxy-glass printed circuit boards. The main board includes the power supply, the combustibles amplifier and alarm circuits and associated controls. A second board is installed above the main board, and is related primarily to the oxygen detection circuit. It is inaccessible while instrument is assembled, except for the two potentiometers which are reached through access holes on main board. (Second board not found in Model 1238)

1. Five miniature adjustment potentiometers are provided on underside of main circuit board, available for user adjustment when case is opened, by use of a small screwdriver:

- Coarse ZERO, used to balance the Wheatstone bridge to zero initially, when a new detector is installed.
- LEL ALARM Threshold, to set the audible alarm to be actuated at a specific meter reading on the LEL range. A typical setting is 20% LEL.
- PPM ALARM Threshold, to set the audible alarm to be actuated at a specific meter reading on the ppm range. 100 ppm would be a typical setting.
- LEL SPAN, is used to set sensitivity or span in LEL range.
- PPM SPAN, is used to set sensitivity in ppm range.

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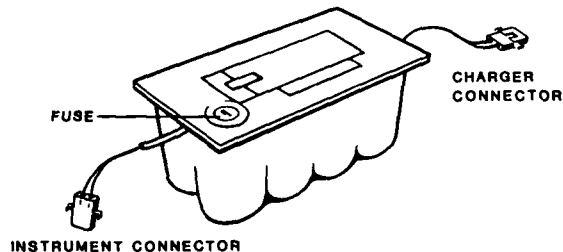


FIGURE 7. STANDARD BATTERY PACK

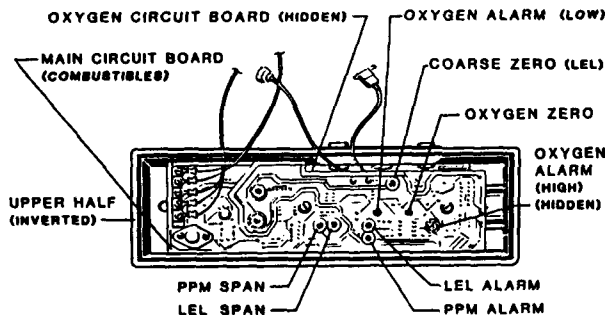


FIGURE 8. CIRCUIT BOARDS WITH ADJUSTMENTS

2. Two miniature adjustment potentiometers are provided on the underside of upper oxygen circuit board, accessible when case is opened and adjustable by use of a small screwdriver through the two holes in main board.

- a) ZERO, to balance the oxygen circuit for zero output while the detector is sampling oxygen-free gas such as nitrogen. This is the control closer to the rear.
- b) ALARM Threshold, to set the oxygen concentration at which the oxygen deficiency alarm is actuated.
- c) The high oxygen alarm (25%), provided on the later models, is factory set, and can be readjusted only when the main board is removed.

I. Sample System

Sample system consists of the flow path, from probe to hose to sample inlet to reaction chamber to pump, to oxygen chamber and exhaust. These components are further described below.

1. Probe consists of a 10" long 1/4" OD plastic tube with a dust filter chamber at the upper end, forming a handle. This filter chamber is of transparent acrylic plastic, so the filter condition can readily be inspected. To replace filter, unscrew filter chamber where it connects to the nickel-plated threaded base.
2. Hose, a 5' flexible polyurethane tube equipped with a male quick-connect coupling with an O-ring seal, to engage inlet fitting of instrument on one end. The opposite end has a threaded fitting onto which the probe is fitted.
3. Inlet fitting, a quick-release female coupling on front of instrument, is screwed directly into reaction chamber. A sintered stainless steel disk is permanently installed within fitting, and serves as a filter to remove dust from incoming sample.
4. Reaction chamber, housing combustibles detector, is an anodized aluminum block with threaded neck and lock nut to retain detector in flow path of sample. An outlet nipple and flexible tube connect chamber to pump.
5. Pump is of the motor driven diaphragm type, with a brushless DC motor having no commutator or sparking contacts. It operates directly from the battery output whenever power switch is on.
6. Oxygen chamber, holding oxygen detector, is an anodized aluminum block with a cavity into which oxygen cell is clamped. An O-ring seal is provided to prevent leakage from chamber, and a spring clamp bar maintains pressure against the seal, but can be swung aside and removed when changing cell.

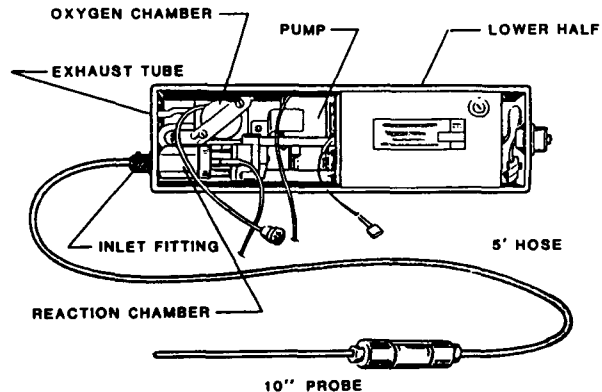


FIGURE 9. SAMPLE DRAWING SYSTEM

J. Charger

A separate battery charger is provided, which plugs into socket in rear of case. Charger provides current at a rate sufficient to recharge batteries overnight. Current tapers down to the point that it can be left connected for several days without damaging battery; however, avoid charge periods longer than 24 hours if possible.

Charger is wired for 115V or for 230AC 50/60 Hz power source (see nameplate for voltage rating of charger supplied with this instrument.)

Later instruments are provided with the No. 49-2133 (49-2134 for 220-240V AC) dual-rate timed charger, which provides a full charge over a 16-hour period, then automatically cuts back to a sustaining rate. An amber light shows that battery is receiving a charge; when complete, the green light indicates that the battery is ready to use. This charger is also available as a replacement charger for any GasTechtor.

K. Continuous Operation

Instrument can be operated continuously from a 12 volt DC source, such as a 12 volt vehicle battery, by use of a Continuous Operation Adapter. This is a power cord with voltage-dropping diodes built in, and with a mating plug to fit charger socket. When connected to instrument and to a 12 volt source, it will carry the load and tend to recharge the battery. It may also be used as a DC charger.

Adapter is furnished with a cigarette lighter plug to fit any negative-grounded vehicle with 12 volt battery. Order Part No. 47-1501.

An adapter for operation from 115V AC is also available. Order Part No. 49-2037.

NOTE

The intrinsic safety rating of the GasTechtor does not apply while being operated from an external power source.

L. Auxiliary Battery Pack

Where sustained battery operation of more than 8 hours is required, an auxiliary battery pack accessory is offered. It is assembled into a housing which clamps to the bottom of the GasTechtor and plugs into charger connection. This extends operation for an additional 8 hours. Order Part No. 49-8054.

M. Sample Line Filter/Trap

For moist or dusty environments, two filter accessories are offered:

1. A glass-bodied filter-trap assembly is offered, to fit instrument inlet and accept the sample hose. This will remove dust and liquid water from incoming sample line. Order Part No. 80-0204.
2. A disposable plastic filter, with water repellent (hydrophobic) element is available for insertion into the sample line at the instrument end. It will block water from passing through the filter and into the instrument, as well as removing particles. Order Part No. 80-0222.

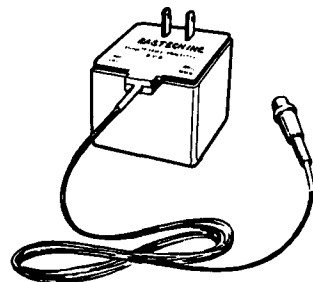


FIGURE 10. BATTERY CHARGER

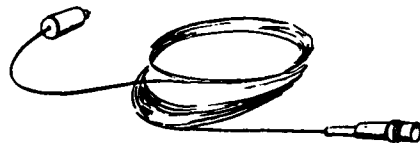


FIGURE 11. CONTINUOUS OPERATION ADAPTER

III. OPERATION

A. Normal Operation

To use instrument, carry out the following steps:

1. Attach hose and probe to instrument by means of the quick release fitting.
2. Put LEL-PPM switch in LEL (out) position, with black indicator showing, and OXY-PPM/LEL switch also in LEL (out) position.

Press POWER switch to turn instrument on, with orange indicator dot showing. Meter will normally rise upscale and pulsing or steady alarm signal may sound. Audible hum of pump will be noticed. Cause of alarm condition (combustibles, oxygen, or both) can be identified by the blinking lights.

4. Press BATTERY CHECK button and note meter reading. If reading is close to or below BATT CK mark on meter, recharge battery.
5. Allow to warm up until meter stabilizes (about a minute). If pulsed oxygen alarm continues to sound, turn OXY CAL potentiometer clockwise to stop it. If the sound is steady, turn the potentiometer counterclockwise. Then, with hose inlet in a gas-free location, turn PPM/LEL ZERO potentiometer to bring meter to "0" indication. (If impossible to reach zero within span of potentiometer, adjust COARSE ZERO. See V.A.2. and 3.)
6. Next put OXY-PPM/LEL switch in the OXY (in) position, so that orange indicator shows. Turn OXY CAL potentiometer to bring meter to the O₂ CAL mark (21%).

As a quick check, gently breathe into hose inlet and allow instrument to sample expired air. Reading should come down to about 16, and alarm should sound at 19.5%. Allow it to return to 21, then put switch back in LEL position.

7. An atmosphere containing more than the normal 21% oxygen content will produce an increased oxygen reading. If instrument is provided with a high oxygen alarm, then it will sound in a steady tone and the amber alarm light will blink when reading reaches or exceeds 25%.
8. Instrument will automatically test for oxygen whenever it is used, and will give pulsed audible and amber light alarm if oxygen content drops to 19.5%. It is not necessary to use instrument with switch in the OXY position unless oxygen measurements are of primary interest. If both abnormal gas conditions exist simultaneously, both lights will blink in their normal pattern, but buzzer will sound continuously.

9. For readings in the 0-100% LEL range, hold hose inlet at point to be tested. Watch meter and observe maximum reading as taken from the upper set of graduations, 0-100% scale. If reading rises above alarm setting (initially 20% LEL), pulsed red light and audible alarm will commence, and will continue as long as reading remains above alarm point. After taking reading, purge instrument with fresh air before turning off.
10. If reading on 0-100% range is imperceptible or very small, use the sensitive range, 0-500 ppm. First allow to warm up in the LEL range, and then push LEL-PPM range switch to put circuit in PPM range (colored indicator shown). Reread carefully with the PPM/LEL ZERO potentiometer.

Because of the very high sensitivity of this range, meter will tend to drift until instrument is thoroughly warmed up. Always let it run for 5 minutes or more, whenever possible, before operating on the PPM range. Take reading immediately after zeroing, and observe maximum deflection as taken from the middle set of graduations, 0-500 PPM scale. Again, alarm will sound whenever reading rises above preset alarm level (initially 100 ppm).

NOTE

Instrument may be equipped with the optional 0-1000 ppm sensitive range, or with some special range. See Title page for information on actual range of instrument as supplied by factory.

IV. INTERPRETATION

A. LEL Range

This range gives readings in terms of nearness to the lower explosive limit (LEL), with 100% corresponding to the LEL of the gas for which the instrument is calibrated.

Detection in this range is by catalytic oxidation on a platinum element, with the heat of oxidation producing a temperature rise and hence a resistance rise of the element. This change is approximately linear up to the LEL provided there is sufficient oxygen in the sample to support combustion.

B. PPM Range

This is a very sensitive range, obtained by amplification of the signal from the catalytic element. Sensitivity is set for a direct reading in ppm of the gas for which the instrument is calibrated.

Even though the sensing element is compensated to minimize the effect of non-combustible gases, a residual effect is still observable at the high sensitivity level. Thus the instrument may need to be rezeroed if exposed to a change in humidity, or to a change in background level of CO₂ or other inert gas.

Other combustible gases will give a response on the PPM range, but will not be direct-reading and will require a calibration curve or correction factor. A few typical conversion factors are included in this manual; refer to Table I, page 33.

C. Oxygen Range (Model 1314 only)

Normal readings will be around 21%, with alarm set about 19.5% for decreasing oxygen. If instrument is provided with a high oxygen alarm, it will sound at 25% on increasing oxygen. However, readings are linear all the way from 0-25%. If readings are to be taken close to zero, as in checking inert gas, first check and adjust zero setting, as described in Sect. V.

D. Abnormal Indications

1. If battery voltage drops below the designed value (about 8.2 volts) the low battery alarm will sound. This is a continuous audible tone. To verify the cause of the alarm, press BATTERY CHECK switch and note that meter reads below check mark. Intensity of alarm sound will increase gradually over a period of a half hour, giving ample warning prior to actual failure.
2. If combustibles range (LEL or PPM) drifts or moves below 0 by 10% or more, the low limit alarm will sound. This is also a continuous tone, and the cause can be recognized by a glance at the meter, in the LEL or PPM range. Downscale meter movement can be caused by incorrect zero adjustment, or by a break in detector or detector wiring.

3. If oxygen cell output declines or deteriorates, as is likely toward the end of cell life, this will produce a reduced reading, and alarm.
4. If oxygen detector is unplugged, or if one of the wires connecting it internally is broken, reading will go to zero, or below, and alarm will come on.
5. On some later models, the steady audible tone sounds when the oxygen reading exceeds 25%. This characteristic is provided to warn against the increased fire hazard due to excess oxygen. It also serves as a warning in case of oxygen cell failure in the high-output mode, which can occur occasionally. It further precludes accidental or intentional incorrect adjustment of the oxygen calibration control to an abnormally high level above 25.

V. CALIBRATION AND ADJUSTMENT

A. Combustibles Calibration

To check and adjust calibration on a known gas sample.

1. Turn instrument on and allow it to warm up and stabilize, preferably for 5 minutes. Be sure batteries are charged sufficiently to read above the check mark.
2. Open instrument case by loosening captive screw at front. Lift upper half of case slightly, move 1/4" to rear to disengage rear clamp; then separate the two halves. Locate COARSE ZERO potentiometer on underside of circuit board (marked "ZERO").
3. Turn external PPM/LEL ZERO control to center of its span. Then turn COARSE ZERO potentiometer to bring meter to zero reading.
4. To calibrate the LEL range, insert sample inlet tube into a vessel or other source of known calibrating gas. Watch meter and note highest reading. If it is incorrect, turn LEL SPAN potentiometer to give desired reading.
5. For PPM calibration, follow same procedure with range switch in PPM position, and use PPM SPAN potentiometer. Before making this adjustment, allow system to warm up and stabilize thoroughly, and zero carefully in the PPM range.

NOTE

In the sensitive ppm range it is important that the humidity of the sample be the same as that of the air used for zero adjustment. If they are different, a significant offset in zero reading may be observed. To overcome this, a humidifier may be used for both zero and calibration tests, providing the calibrating gas is one that will not be absorbed in water. The GasTech Calibration Kit is supplied complete with humidifier and accessories for this effect. See Appendix A.

6. If zero cannot be adjusted, or if reading cannot be set high enough, replace detector.

B. Combustibles Alarm Threshold

The reading at which the alarm is actuated in each range can be set by use of the corresponding ALARM Threshold potentiometer.

To Set:

1. With instrument in range to be set, turn PPM/LEL ZERO adjust to bring meter to desired alarm setting. It may be necessary to use the COARSE ZERO to reach this point, in LEL range.
2. Turn corresponding (LEL or PPM) ALARM Threshold potentiometer to the point where alarm just operates. Clockwise rotation will lower alarm setting. Verify setting by turning PPM/LEL ZERO control to bring meter indication into and out of alarm zone.
3. When the combustibles Alarm Threshold has been satisfactorily set, readjust the zero potentiometers to establish a zero meter reading as in A.3 above.

C. Oxygen Zero Adjustment

The following steps should be carried out with OXY-PPM/LEL switch on OXY (in) position. To check and adjust zero on a known oxygen-free sample:

1. While instrument case is open, identify ZERO potentiometer, which is located on oxygen (upper) circuit board and which can be reached through the rear-most of the two clearance holes in the main circuit board.
2. Allow instrument to sample a known oxygen-free sample, such as nitrogen, argon, or helium.
3. Watch meter carefully. If reading does not go exactly to zero, adjust it by turning ZERO potentiometer. Counterclockwise rotation will decrease reading.
4. If zero adjustment cannot be made, have oxygen cell reactivated.
5. After zero adjustment has been completed, return hose inlet to normal atmospheric air. Readjust OXY CAL control as necessary to bring meter reading to 21.
6. If reading cannot be set high enough, have oxygen cell reactivated.

D. Oxygen Alarm Threshold

The reading at which the oxygen alarm is actuated can be set by use of the ALARM Threshold potentiometer.

To Set:

1. Turn OXY CAL to bring meter to desired alarm setting.
2. Locate oxygen alarm threshold potentiometer, on oxygen circuit board, which is accessible through the hole adjacent to the LEL Alarm adjustment.
3. Turn ALARM Threshold potentiometer to the point where alarm just operates. Clockwise rotation will raise alarm setting. Verify setting by turning OXY CAL control to bring meter into and out of alarm zone.
4. When the oxygen Alarm Threshold has been satisfactorily set, readjust the OXY CAL potentiometer to bring the meter reading to 21% (O₂ CAL).

E. Oxygen High Alarm (25%)

This alarm point is factory set and generally need not be changed. It can be readjusted to some other level, by trial, but this can only be done when the main board is loosened and pulled aside (see Section VI.D. steps 1-6). The high alarm potentiometer is the one closest to the rear, without an access hole.

VI. MAINTENANCE

A. Batteries

1. Check battery voltage periodically by pressing BATTERY CHECK switch. Recharge before voltage reaches minimum.

When connecting charger, always follow these steps:

- a) Confirm that the plug is inserted in the correct way, with the THIS SIDE UP label upwards. The socket is polarized, with the pins offset below the centerline, but can sometimes be forced on the wrong way, particularly if it has become worn with use.
- b) Verify that a charge is actually entering battery. To do this, turn instrument on and check meter reading while BATTERY CHECK button is pressed. Observe reading while charger is plugged and unplugged at wall socket. If reading increases when charger is connected, and decreases when unplugged, battery is receiving a charge. If no change is observed, then probably charger or power circuit is at fault. If charger is defective, return it for repair or replacement.

After verifying that instrument is accepting a charge, turn instrument switch off. Do not attempt to charge while instrument is turned on.

2. If sufficient voltage cannot be obtained after charging, open instrument and:

- a) Check voltage output with a voltmeter, between red and black wires (unplug connector to gain access to pins). Voltage should be about 8.5 volts.

If voltage is acceptable, but volt check reading is too low, meter or switch could be at fault. If voltage is too low, battery is at fault.

- b) If no output voltage can be obtained, check fuse by unscrewing recessed cap, marked "FUSE", and removing fuse. It can be checked visually or with an ohmmeter. If burned out, replace with a new one, but be sure to attempt to identify the cause of the overload or short circuit. Fuse must be type 3AG-1A.
- c) If battery voltage is too low, and cannot be brought up by overnight charging, it probably needs replacement. To remove, take out the two screws holding it to bottom of case, and unplug black and orange wire connector at charging end.

3. If the replaceable cell battery pack has been installed and is found defective, open the pack and check the voltage of each individual cell with a voltmeter. Remove two screws on the top of the pack with a 1/8" Allen wrench. The lid is spring loaded and may be held down by hand to ease the screw removal. Carefully remove the lid and the individual cells.

- a) The cells supplied are the Stock No. 49-1501 rechargeable D-size nickel-cadmium type, 1.5-4.0 AH, and when charged, measure about 1.35 volts. Discard and replace faulty cells.
- b) Examine the battery cavity and carefully clean out all foreign substances. Reinsert the cells into the pack in accordance with the diagram on the lid. (Negative end to springs, button end to rivets.) Leave the proper space open for the fuse cartridge.
- c) Place the lid onto the cells, press down firmly and insert screws. The convoluted case will assure cell alignment. Tighten the screws snugly, do not overtorque. It may be necessary to spring sides of instrument case apart slightly to clear battery lid.
- d) Join all loose connectors, reassemble instrument and charge battery as required.
- e) If normal operation from disposable batteries is desired, use the Stock No. 45-8052 battery pack instead of the 49-8051 pack. The spring-loaded top is held down by two knurled thumb screws. When replacing, tighten both screws at the same time to assure even compression of the springs and proper battery contact. Duracell® type disposable batteries are recommended for a proper fit.

This battery pack has no charger connection, so there is no danger of inadvertent charging of disposable cells. It can be used with rechargeable cells, but they must be charged separately.

*Duracell is a trade name for Duracell Inc., Bethel, CT 06801.

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B. Combustibles Detector

1. Sensor assembly may require replacement if:
 - a) Meter cannot be set to zero within range of ZERO potentiometer. (Including COARSE ZERO)
 - b) Meter cannot be set to desired level within range of SPAN Adjust, either range.
2. To replace detector:
 - a) Open instrument case.
 - b) Loosen and remove the red, green and white wires at terminals on main circuit board, noting color coding.
 - c) Unscrew knurled retaining cap at reaction chamber.
 - d) Pull out original detector and install new one, being sure that O-ring is in place, under flange of detector.
 - e) Connect wires to terminals, turn power on, and adjust COARSE ZERO as in Section V.A.3.

C. Oxygen Detector

1. Oxygen sensor assembly may require repair if:
 - a) Meter cannot be set to desired level within range of OXY CAL Adjust.
 - b) Meter cannot be set to zero within range of ZERO potentiometer.
2. If oxygen sensor assembly requires repair, it should be sent to factory for reactivation. Alternatively, a complete new detector can be ordered, on an exchange basis.

Oxygen cell is an electrochemical device similar to a battery, which gradually depletes itself, regardless of usage of the cell. It requires periodic reactivation, consisting of replacement of the electrolyte and the membrane, plus cleaning and inspection of the electrodes. This is most economically done at the factory.

New and reactivated oxygen cells carry a factory guarantee based on length of time from date of original shipment, and are date-coded. Cells returned for reactivation are inspected and in-warranty cells are tested for operability. Any that fail prematurely receive a warranty allowance.

To replace oxygen sensor:

- a) Open instrument case. Locate oxygen cell.
- b) Swing retainer clamp clockwise and remove it to release cell.
- c) Tilt cell upward and pull it out of case. Unplug cell wire at socket.
- d) Reinstall new or newly reactivated cell in same position. Before installing, remove protective seal from face of cell, and shake out any drops of water that may be found under seal.

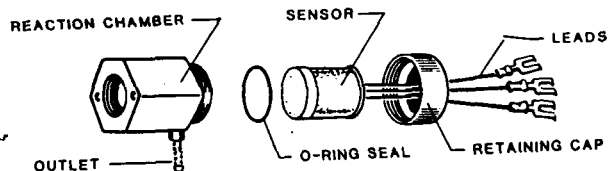


FIGURE 12. COMBUSTIBLES DETECTOR

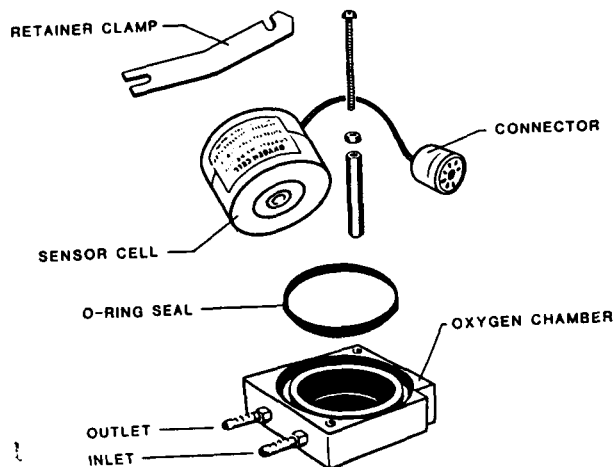


FIGURE 13. OXYGEN DETECTOR

D. Meter

If meter is damaged, it can be removed for repairs or replacement, as follows:

1. With upper half of instrument removed from lower half and inverted, loosen internal lock nuts from POWER and BATTERY CHECK switch bushings and PPM/LEL ZERO potentiometer bushing.
2. Remove external lock nut from PPM/LEL ZERO potentiometer. (First remove knob, if one is used.)
3. Remove external face nuts from switch bushings.
4. Remove three screws holding circuit board into case.
5. Remove two nuts from meter studs.
6. Pull circuit board out of case as far as connecting wires permit.
7. Lift out meter.

E. Buzzer

If buzzer fails, it can be removed by first taking out circuit board (Steps D.1-D.6) and unsoldering red and black wires at board. Then remove retaining screws and nuts.

Note: Before removal, first verify that buzzer is actually defective. Connect to a 6 volt battery (Red +, Black -). A good buzzer will give a steady tone.

F. Circuit Board

Main circuit board can be removed by steps D.1-D.6, plus E, plus disconnection of pump and detector wires at terminals. This allows the board to be pulled out of the case and put aside for access to the oxygen circuit board. However, it will remain connected to the oxygen board until the interconnecting wires are unsoldered.

Preferably, both boards will be removed as a unit, and returned to factory for repairs. To complete removal, loosen internal lock nuts, remove external lock nuts from remaining controls, and take off the hex plastic spacers which hold oxygen board in place.

G. Filter

The filter element in Inlet fitting is pressed in but can be pushed out and replaced. When necessary, the complete fitting can be replaced.

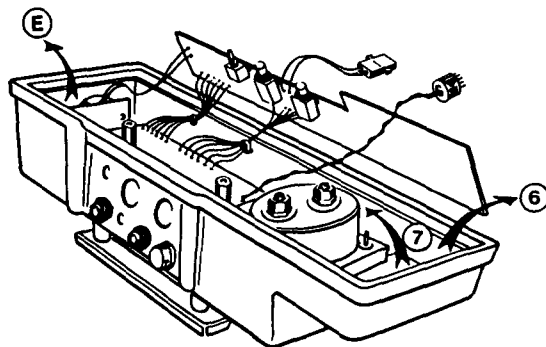
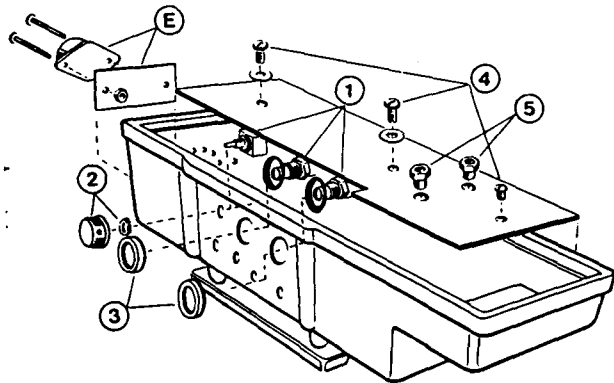


FIGURE 14. METER, BUZZER AND CIRCUIT BOARD REMOVAL / REPLACEMENT

H. Pump

Pump used is a diaphragm type, driven by a brushless DC motor. It should have long life, several years in normal operation, but it may lose efficiency if dirt is drawn in and collects under the valves. Verify proper pump operation periodically by taking a sample and observing time for initial gas response to occur. This should be within 5 seconds for a 10' hose. It may also be checked with the flowmeter provided as a calibration accessory. Flow should be 2.0 cfm or greater.

If pump needs servicing, it can be removed by taking out the clamp retaining screw which extends through case bottom. Pump can be returned for repair on an exchange basis or it can be disassembled and cleaned. Replacement pump head assemblies and diaphragms are available.

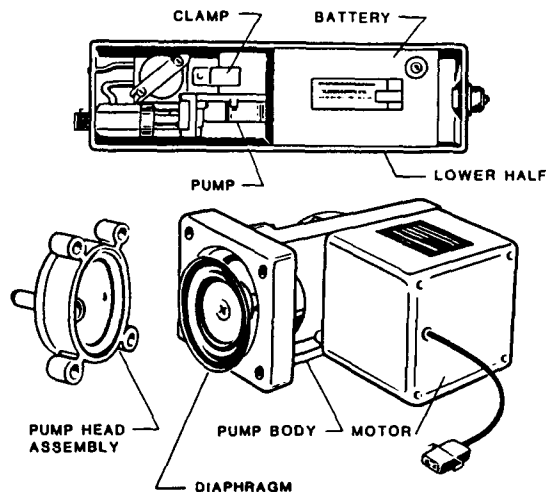
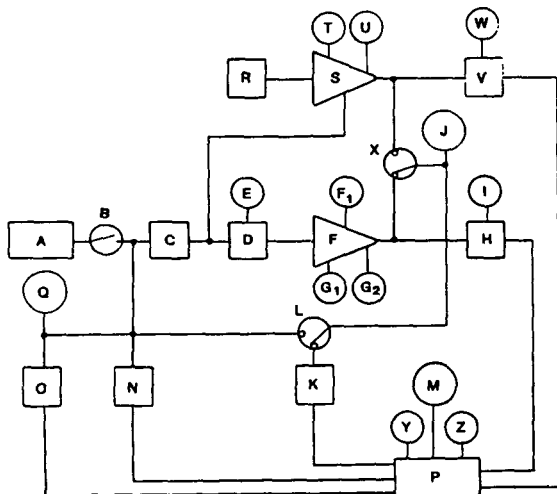


FIGURE 15. PUMP ASSEMBLY FOR MODEL 1314

VII. CIRCUIT DESCRIPTION

Referring to block diagram:



- A. Battery pack, rechargeable, nickel-cadmium, giving 8 hours of operation.
- B. On-off switch, push on/push off.
- C. Voltage regulator, input 8.0 - 10 volts, output 6.0 volts \pm .05.
- D. Combustibles detector, with active (catalytic) and reference (non-catalytic) elements, forming half of Wheatstone bridge measuring circuit. Two fixed resistors complete bridge.
- E. Zero Adjust, a potentiometer in the fixed side of the bridge used to bring the bridge to balance in the absence of gas. Two potentiometers are used, a coarse and fine adjustment.
- F. Amplifier, Signal differential, to increase output of bridge to a suitable level for indication and alarm actuation.
- F1. Range switch, alternate-action pushbutton, to select LEL or PPM ranges by change of amplifier gain.

- G. Span Adjust, to set meter reading to desired value for a known sample. Two adjusters, G1 and G2, serve PPM and LEL ranges respectively.
- H. Alarm switching circuit, to turn on combustibles alarm signal when amplifier output reaches a predetermined point.
- I. Alarm threshold adjustment, to set the point at which combustibles alarm is actuated. (One each for LEL and PPM ranges)
- J. Meter, reads output of amplifier, in units of percent explosibility, in PPM, or in percent oxygen depending upon range.
- K. Malfunction alarm switching circuit, turns on alarm due to amplifier output below zero.
- L. Batt. Test switch, to connect meter as a voltmeter momentarily, to check battery voltage.
- M. Buzzer, a solid-state electronic sounder which delivers a continuous tone when energized.
- N. Multi-Vibrator, an oscillating circuit to give a pulsating signal to buzzer, as an identification of alarm condition.
- O. Voltage-sensing circuit, to detect low battery voltage and actuate alarm signal.
- P. Logic circuit, to accept signals of various types and direct them to buzzer circuit.
- Q. Pump, vibratory diaphragm type, to draw sample continuously through sample system and over detector.
- R. Oxygen Sensor, which supplies a signal voltage proportional to oxygen concentration.
- S. Amplifier, oxygen, to increase output of sensor to a suitable level for indication and alarm actuation.
- T. Zero, oxygen, a potentiometer used to balance output of amplifier to zero in the absence of oxygen.
- U. Span, oxygen, a potentiometer used to set gain of amplifier to give desired sensitivity on a known calibrating sample.
- V. Alarm switching circuit to turn on oxygen alarm signal when amplifier output drops or rises to a preset point.
- W. Alarm threshold adjustment, to set the point at which oxygen alarm comes on. (One each for falling and rising alarm.)
- X. Range switch, to select range for readout of meter, either oxygen or combustibles.
- Y. Alarm light, oxygen, which blinks on and off whenever system is in oxygen alarm condition.
- Z. Alarm light, LEL, which blinks on and off whenever system is in combustibles alarm condition.

VIII. PRECAUTIONS AND NOTES ON OPERATION

A. Heated Samples

When sampling spaces such as hot tanks that are warmer than the instrument, remember that condensation can occur as the sample passes through the cooler sample line. Water vapor condensed in this way can block the flame arrestor and interfere with pump operation.

If heated hydrocarbon vapors of the heavier hydrocarbons (flash point 90°F or above) are present, they may also condense in the sample line and fail to reach the filament. Thus an erroneous low reading may be obtained.

B. Filament Poisoning

Certain substances have the property of desensitizing the catalytic surface of the platinum filament. These substances are termed "catalyst poisons" and can result in reduced sensitivity or in failure to give a reading on samples containing combustible gas. The most commonly encountered catalyst poisons are the silicone vapors, and samples containing such vapors even in small proportions should be avoided.

Occasional calibration checks on known gas samples are desirable, especially if the possibility exists of exposure to silicones. Where persistent poisoning action occurs, special silicone-resistant elements are available.

C. Other Gases and Vapors

The instrument is designed and calibrated specifically for the gas specified in the original order. It can be recalibrated and used on other gases and vapors, by proper use of the calibration control while sampling a known gas-air mixture.

D. Rich Mixtures

When sampling rich mixtures, on the 100% LEL range, the following instrument action may be expected:

1. mixtures up to 100% L.E.L. - Reading on scale
2. mixtures between L.E.L. and Upper Explosive Limit (U.E.L.) - Readings at top of meter
3. mixtures above U.E.L. - As sampling continues, the meter first goes to top of scale, then comes back down on scale. Very rich mixtures will give close to a zero reading.

E. Oxygen Deficient Mixtures

Samples which do not have the normal proportion of oxygen may tend to read low, as there is not enough oxygen to react with all combustible gas present in the sample. As a general rule, samples containing 10% oxygen or more have enough oxygen to give a full reading on any combustible gas sample up to the L.E.L.

F. Arson Investigation

Flammable liquids (gasoline, kerosene or paint solvent) are often used in starting intentional fires. Investigation of such fires can be greatly aided if the presence and location of such liquids can be determined at the site, as soon as possible after the fire is extinguished. The Hydrocarbon Surveyor can be of great assistance in making this determination.

In testing for residual flammable liquids, look for places where the liquid could have been trapped and where it might remain even after the fire. Naturally, if the entire structure has been consumed there is little likelihood of any liquid or vapors remaining. Conversely, the earlier the fire has been extinguished, the greater the chance of finding significant amounts of liquid remaining.

In checking for residual volatile liquids, set the instrument up in accordance with the preceding instructions, allowing it to run for at least 5 minutes, in the LEL range. Then turn to ppm range and balance zero carefully immediately before taking the test.

Hold end of hose or probe at point where vapors may be present, and watch meter carefully for any sign of a deflection. Check at joints or cracks between boards, for example under baseboards or plates in contact with flooring. Pry boards up to form a small crack where hose or probe may be inserted. Check also under unburned portions of rug or upholstery, or any point where liquid might logically have soaked in and remained.

If a positive indication is obtained, trace it to the point of maximum reading. This is the point where samples should be taken for further lab analysis.

IX. PARTS LIST

Stock No.	Description
06-5012	Hose, Polyurethane, inlet, 5'
07-6010	O-Ring Seal, hose (probe end)
07-6011	O-Ring Seal, hose (instrument end)
07-6014	O-Ring Seal, inlet fitting
07-6115	O-Ring Seal, combustibles detector
07-6216	O-Ring Seal, oxygen cell cavity
17-0434	Inlet fitting w/filter
33-1016	Filter disk only, inlet fitting
30-0016	Pump, rotary DC
30-0340	Pump head, replacement
30-0342	Pump valves, replacement, set of 2
43-4140	Fuse, battery, JAG 1A
45-8051	Battery Pack, replaceable, rechargeable cell, less batteries
45-8052	Battery Pack, replaceable, disposable cell, less batteries
49-1201	Battery, alkaline size D disposable (for use in 45-8052)
49-1501	Battery, Ni-Cad, rechargeable (for use in 45-8051)
49-1571	Battery Pack, encapsulated with Ni-Cad batteries
49-2033	Battery Charger, 115 volts, for Ni-Cad batteries
49-2133	Battery Charger, 115 volts, dual-rate time-controlled
49-2034	Battery Charger, 230 volts, for Ni-Cad batteries
49-2134	Battery Charger, 230 volts, dual-rate time-controlled (user to provide AC plug)
49-8051	Battery Pack, replaceable, rechargeable cell, with batteries
50-1226	Meter, PPM/LEL Scale (1238)
50-1229	Meter, PPM/LEL/O ₂ Scale (1314)
52-1005	Buzzer
61-0120	Detector Ass'y, wired for Surveyor
65-0601	Oxygen cell, new
65-0601E	Oxygen cell, reactivated (exchange)
71-0115	Instruction Manual, Model 1314/1238
80-0150	10" Probe
80-0204	Moisture Trap
80-0222	Filter, hydrophobic

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TABLE I

Relative Response of New GasTector to Various Gases (Referenced to Hexane or Toluene)

GAS	LEL Scale			PPM Scale		
	LEL in %	Conversion Factor Hexane	Conversion Factor Toluene	TLV in ppm*	Conversion Factor Hexane	Conversion Factor Toluene
Acetone	2.5	0.65	0.63	1000	1.55	1.30
Acrylonitrile	3.0	-	-	1	1.31	1.1
Benzene	1.3	0.87	0.84	10	1.11	0.93
Butadiene	2.0	0.88	0.85	1000	2.0	1.7
Chloroform	-	-	-	50	8.0	6.7
Carbon Monoxide	12.5	0.60	0.58	50	4.4	3.7
Ethyl Acetate	2.0	0.81	0.79	400	1.61	1.35
Ethyl Alcohol	3.3	0.60	0.58	1000	2.0	1.67
Formaldehyde***	7.0	1.96	1.9	3	7.4	6.2
Heptane	1.05	1.03	1.00	500	0.98	0.82
Hexane	1.1	1.00	0.97	500	1.00	0.84
Hydrogen Sulfide	4.0	1.96	1.9	20	5.0	4.2
Methyl Chloroform	-	-	-	350	3.7	3.1
Methyl Ethyl Ketone	1.4	0.84	0.81	200	1.3	1.09
Methylene Chloride	14.0	2.78	2.7	500	4.4	3.7
Methane	5.0	0.42	0.41	-	2.4	2.0
Pentane	1.5	0.71	0.69	1000	1.81	1.52
Perchloroethylene	-	-	-	100	4.5	3.8
n-Propyl Acetate	1.7	0.77	0.75	200	1.36	1.14
n-Propyl Alcohol	2.2	0.80	0.78	200	1.52	1.28
Styrene	1.1	1.24	1.2	100	1.31	1.1
Toluene	1.2	1.03	1.00	200	1.19	1.00
Trichloroethylene	8.0	0.59	0.57	25	4.4	3.7

Please note, that response varies from one sensor to another and the relative response of a sensor can change with age, so these data should be used for estimation purposes only.

Assuming an instrument calibrated directly for hexane or toluene, but used to observe a different gas, the equivalent response in LEL (or ppm) for that gas is secured by multiplying the observed LEL (or ppm) reading by the LEL (or ppm) scale conversion factor.

* Values from National Fire Protection Association, NFPA 325M, 1984.

** Values from Federal Register 40,23072, May 28, 1975 and current as of November 1979, except for trichloroethylene where a CAL/OSHA November 1980 value is listed.

*** Methanol free.

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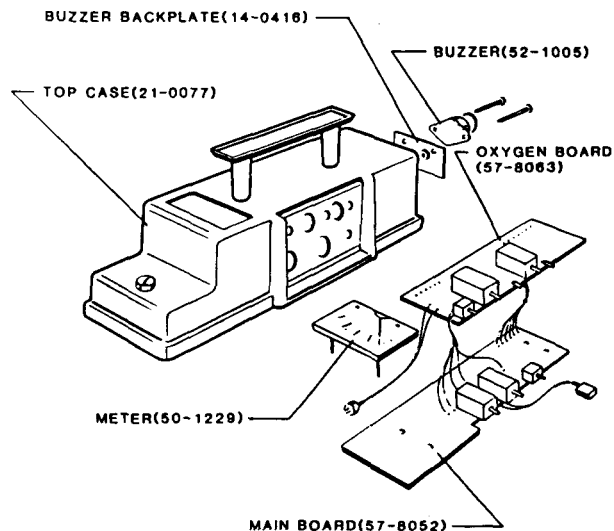


FIGURE 16. PARTS LOCATION-TOP HALF

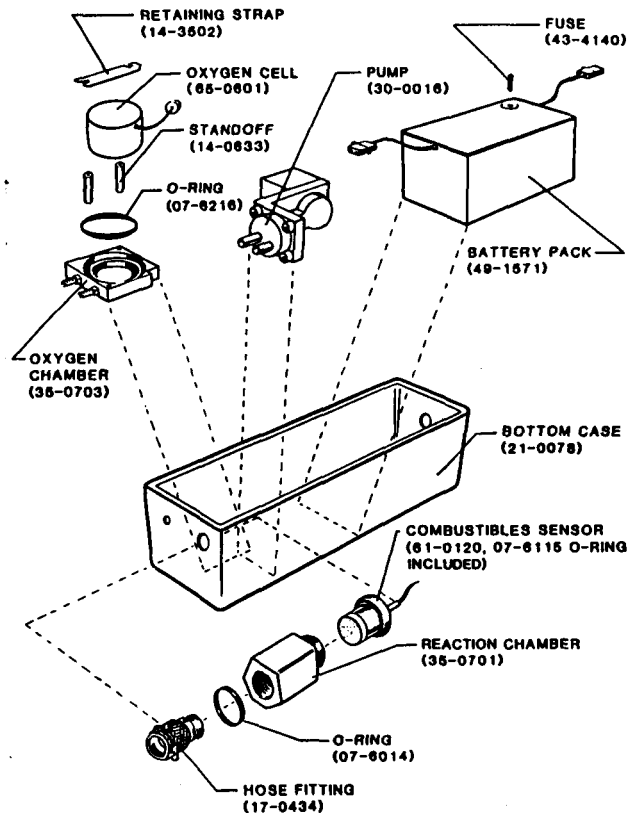
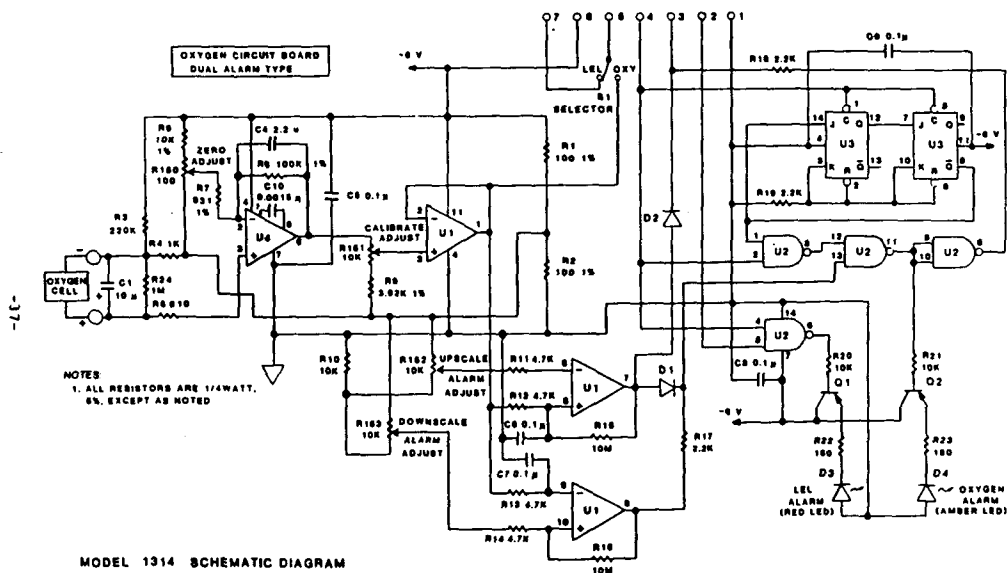
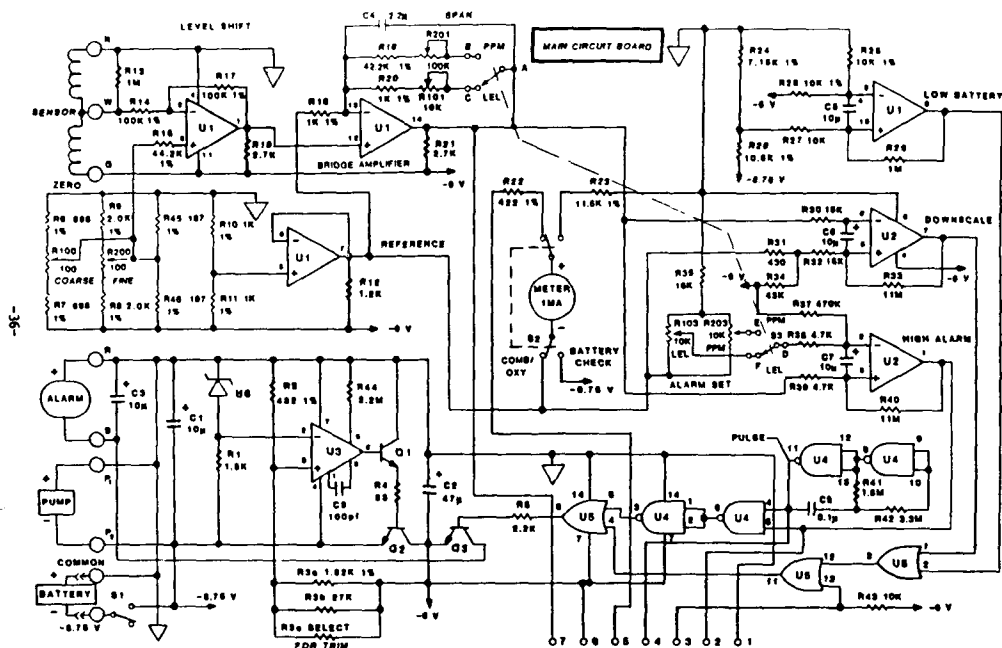


FIGURE 17. PARTS LOCATION-LOWER HALF



MODEL 1314 SCHEMATIC DIAGRAM

GASTECH CALIBRATION KIT

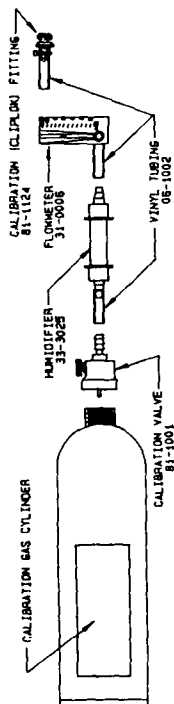
A convenient calibration kit is available for accurate on-site adjustment, without the use of special tools or fixtures. The 81-0222 Calibration Kit is designed expressly for use with the Models 1314 and 1238, and consists of the following:

20-0110	Padded carrying case
81-1124	Calibration fitting
31-0006	Flowmeter
33-3025	Humidifier
81-1001	Calibration gas dispensing valve
81-0007	Calibration gas cylinder 40% LEL hexane
81-0091	Calibration gas cylinder 400 PPM toluene
06-1002	Vinyl plastic tubing

To check and adjust calibration on a known gas sample, assemble the various calibration kit items in accordance with GasTech Drawing 2127-A14, but do not attach the calibration system to the instrument or gas cylinder until the corresponding steps are completed:

1. Turn instrument on and allow it to warm up and stabilize, preferably for 5 minutes. Be sure batteries are charged sufficiently to read above the BATT CK mark.
2. Add water to humidifier, thoroughly moistening the glass wool, and shake out any excess liquid.
3. Open instrument case by loosening captive screw at front. Lift upper half of case slightly, move 1/4" to rear to disengage rear clamp; then separate the two halves. Locate coarse ZERO potentiometer on underside of circuit board (marked "ZERO").
4. Turn external PPM/LEL ZERO control to center of its span. Then turn coarse ZERO potentiometer to bring meter to zero reading.
5. To calibrate in PPM range, press the LEL-PPM switch to the PPM range. Attach the calibration fitting, flowmeter and the humidifier to the instrument. Readjust zero as necessary with the external PPM/LEL ZERO adjustment. Note flow reading on flowmeter.
6. Connect PPM-range cylinder to humidifier inlet and slowly open the calibration valve until the same flow is indicated on the flowmeter as observed previously. Watch meter and note highest reading. If it is not the same as indicated on the calibration cylinder, turn PPM SPAN potentiometer to give desired reading.
7. For LEL calibration, follow steps 1 through 5 with range switch in LEL position, and use LEL-range cylinder and the LEL SPAN potentiometer. However, humidifier is not necessary and may be omitted.
8. If zero cannot be adjusted, or if reading cannot be set high enough, replace detector.
9. Disassemble and store the calibration kit.

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CALIBRATION PROCEDURE

1. PPM RANGE
2. TURN ON INSTRUMENT. ALLOW TO WARM UP AND ADJUST ZERO IN NORMAL WAY.
3. ADD A FEW DROPS OF WATER TO GLASS WOOL PACKING INSIDE HUMIDIFIER.
4. SHAKE HUMIDIFIER TO MOISTEN GLASS WOOL.
5. ATTACH CALIBRATION FITTING TO CALIBRATION INLET.
6. ATTACH FLOWMETER TO CALIBRATION FITTING. INLET HUMIDIFIER TO FLOWMETER INLET, AS SHOWN.
7. COUPLE CLIP ON FLOWMETER TO INSTRUMENT INLET.
8. READJUST ZERO AS REQUIRED AFTER INSTRUMENT STABILIZES.
9. CONNECT CALIBRATION VALVE TO PPM-RANGE CYLINDER.
10. OPEN VALVE SLIGHTLY TO PRODUCE A SMALL FLOW.
11. COUPLE VALVE OUTLET TO HUMIDIFIER INLET.
12. SLOWLY OPEN VALVE UNTIL SAME FLOW IS OBSERVED IN S. MATCH METER AS GAS ENTERS INSTRUMENT. OBSERVE HIGHEST READING.
13. COMPARE READING WITH KNOWN GAS CONCENTRATION ON CYLINDER.
14. IF NOT CORRECT, ADJUST CALIBRATION AS SHOWN IN CALIBRATION SECTION.
15. IF NOT CORRECT, ADJUST CALIBRATION AS SHOWN IN CALIBRATION SECTION.
16. TURN OFF VALVE AND DISCONNECT CALIBRATION COMPONENTS.
17. LEL RANGE
18. USE SAME PROCEDURE AS ABOVE, BUT IN LEL RANGE. HOWEVER, OMIT HUMIDIFIER AS IT IS NOT NECESSARY IN LEL RANGE. USE LEL RANGE CYLINDER.

GASTECH INC. NEWARK, CALIFORNIA 94380	
TITLE: CALIBRATION GAS-FLOW DIAGRAM	
SCALE: NONE	DATE: 5-23-76
BY: KHC	CHKD BY: DJW
REV: 1	REV: 1
DO NOT SCALE DRAWING	
TOLERANCES & FINISHES: DIMENSIONS & FINISHES: 0.015" UNLESS OTHERWISE NOTED	
FRACTIONS: 1/16" 1/8" 1/4" 3/8" 1/2" 5/8" 3/4" 1" 1 1/4" 1 1/2" 1 3/4" 2" 2 1/4" 2 1/2" 2 3/4" 3" 3 1/4" 3 1/2" 3 3/4" 4" 4 1/4" 4 1/2" 4 3/4" 5" 5 1/4" 5 1/2" 5 3/4" 6" 6 1/4" 6 1/2" 6 3/4" 7" 7 1/4" 7 1/2" 7 3/4" 8" 8 1/4" 8 1/2" 8 3/4" 9" 9 1/4" 9 1/2" 9 3/4" 10" 10 1/4" 10 1/2" 10 3/4" 11" 11 1/4" 11 1/2" 11 3/4" 12" 12 1/4" 12 1/2" 12 3/4" 13" 13 1/4" 13 1/2" 13 3/4" 14" 14 1/4" 14 1/2" 14 3/4" 15" 15 1/4" 15 1/2" 15 3/4" 16" 16 1/4" 16 1/2" 16 3/4" 17" 17 1/4" 17 1/2" 17 3/4" 18" 18 1/4" 18 1/2" 18 3/4" 19" 19 1/4" 19 1/2" 19 3/4" 20" 20 1/4" 20 1/2" 20 3/4" 21" 21 1/4" 21 1/2" 21 3/4" 22" 22 1/4" 22 1/2" 22 3/4" 23" 23 1/4" 23 1/2" 23 3/4" 24" 24 1/4" 24 1/2" 24 3/4" 25" 25 1/4" 25 1/2" 25 3/4" 26" 26 1/4" 26 1/2" 26 3/4" 27" 27 1/4" 27 1/2" 27 3/4" 28" 28 1/4" 28 1/2" 28 3/4" 29" 29 1/4" 29 1/2" 29 3/4" 30" 30 1/4" 30 1/2" 30 3/4" 31" 31 1/4" 31 1/2" 31 3/4" 32" 32 1/4" 32 1/2" 32 3/4" 33" 33 1/4" 33 1/2" 33 3/4" 34" 34 1/4" 34 1/2" 34 3/4" 35" 35 1/4" 35 1/2" 35 3/4" 36" 36 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SERVICE POLICY

GasTech Inc. maintains an instrument service facility at the factory. Some GasTech distributors also have repair facilities; however, GasTech assumes no liability for service performed by other than GasTech personnel. Should your instrument require non-warranty repair, you may contact the distributor from which it was purchased, or you may contact GasTech directly.

If GasTech is to do the repair work for you, you may send the instrument, prepaid, to GasTech Inc., 8445 Central Avenue, Newark, CA 94560, Attn: Service Department. Always include your address, purchase order number, shipping and billing information and a description of the defect as you perceive it. If you wish to set a limit to the authorized repair cost, state a "not to exceed" figure. If you must have a price quotation before you can authorize the repair cost, so state, but understand that this involves extra cost and extra handling delay. GasTech's policy is to perform all needed repairs to restore the instrument to full operating condition, including reactivation of all out-of-warranty electrochemical cells.

To expedite the repairs operation, it is preferable to call in advance to GasTech Instrument Service, (415) 794-6200, obtain a Return Authorization Number (RA#), describe the nature of the problem and provide a purchase order number.

If this is the first time you are dealing directly with the factory, you will be asked to provide credit references or prepay, or authorize COD shipment.

Pack the instrument and all its accessories (preferably in its original packing). Enclose your Purchase Order, shipping and billing information, RA#, and any special instructions.

GasTech Inc.
Standard Warranty
Gas Detection Instruments

We warrant gas alarm equipment manufactured and sold by us to be free from defects in materials, workmanship and performance for a period of one year from date of shipment to ultimate user. Any parts found defective within that period will be repaired or replaced, at our option, free of charge, f.o.b. factory. This warranty does not apply to those items which by their nature are subject to deterioration or consumption in normal service, and which must be cleaned, repaired or replaced on a routine basis. Such items may include:

- a) Lamp bulbs and fuses
- b) Pump diaphragms and valves
- c) Absorbent cartridges
- d) Filter elements
- e) Batteries
- f) Most catalytic and electrochemical sensors are covered by a separate warranty of 6, 12, or 24 months.

Warranty is voided by abuse including rough handling, mechanical damage, alteration or repair procedures not in accordance with instruction manual. This warranty indicates the full extent of our liability, and we are not responsible for removal or replacement costs, local repair costs, transportation costs or contingent expenses incurred without our prior approval.

GasTech Inc.'s obligation under this warranty shall be limited to repairing or replacing any product which GasTech Inc. Material Review Board examination shall disclose to its satisfaction to have been defective. To receive warranty consideration, all products must be returned to GasTech Inc. at its manufacturing facilities with transportation charges prepaid.

This warranty is expressly in lieu of any and all other warranties and representations, expressed or implied, and all other obligations or liabilities on the part of GasTech Inc. including but not limited to, the warranty of fitness for a particular purpose. In no event shall GasTech Inc. be liable for direct, incidental or consequential loss or damage of any kind connected with the use of its products or failure of its product to function or operate properly.

This warranty covers instruments and parts sold (to users) only by authorized distributors, dealers and representatives as appointed by GasTech.



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APPENDIX G-10

CALIBRATION AND MAINTENANCE OF THE RADIATION ALERT MONITOR

OPERATION MANUAL FOR THE RADIATION ALERT MONITOR 4

INSTRUCTIONS

To operate the Monitor 4, first install a fresh 9 volt alkaline battery (NEDA #1604A or equivalent). The battery compartment is located at the bottom of the back of the instrument and is accessible by pushing down on the finger grip provided. Snap in the battery and slide the cover back into position. The battery life is up to 2,000 hours at normal background radiation levels. NOTE: Always turn the Monitor 4 off before installing the battery.

The Monitor 4 senses ionizing radiation by means of a GM (Geiger-Mueller) tube with a thin mica end window. The tube is fully enclosed inside the instrument. When a ray or particle of ionizing radiation strikes the tube, it is sensed electronically and displayed by a flashing count light on the instrument face. About 5 to 25 of these counts, at random intervals, can be expected every minute from naturally occurring background radiation, depending on your location and altitude. When the switch is in the AUDIO position, the instrument will also beep each time a ray or particle of ionizing radiation strikes the tube.

Be sure the range switch is in the appropriate position when using the instrument. When the range switch is in the x1 (times one) position, the meter reads from 0 to .5 mR/hr or 0 to 500 CPM (counts per minute) as indicated on the meter scale. In the x10 position the reading is multiplied by 10, indicating levels up to 5 mR/hr or 5000 CPM. In the x100 position the reading is multiplied by 100, indicating up to 50 mR/hr or 50,000 CPM (approximately 2,500 times background levels).

This model of the Monitor 4 has an anti-saturation circuit good to at least 5R/hr.

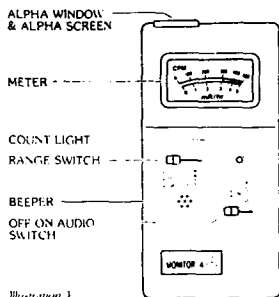


Illustration 1

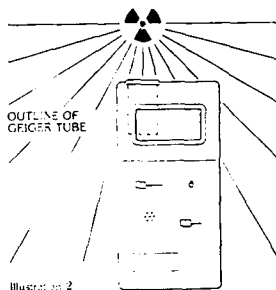


Illustration 2

PRECAUTIONS:

NOTE: Before using this instrument the user must determine the suitability of the product for his or her intended use. The user assumes all risk and liability connected with such use.

1. Handle your Monitor 4 carefully, as you would a camera.
2. Avoid exposing the Monitor 4 to liquids, moisture, and corrosive gases; also avoid extreme temperatures or direct sunlight (i.e. car dashboards) for extended periods.
3. We recommend that you remove the battery to prevent leakage if you don't intend to use the Monitor 4 for a month or more.
4. The mica end window of the Geiger tube can be easily damaged if struck directly. Therefore, do not insert any pointed object through the alpha screen.
5. To avoid contaminating the Monitor 4, do not touch the instrument to the surface being tested.
6. This instrument may be sensitive to radio frequency fields, microwave fields, electrostatic fields, and magnetic fields, and may not operate properly in such fields.
7. Since the Monitor 4 has semiconductors in its circuitry, it is susceptible to EMP (electromagnetic pulse) and may be rendered inoperable by an atomic detonation. The government has classified information on the effects of EMP, therefore we have not been able to ascertain what distance from an atomic blast would be considered safe for semiconductor circuitry.

SERVICE AND CALIBRATION:

The Monitor 4 has proven to be a very reliable instrument. Should it ever need servicing outside of our warranty, please contact us for a return authorization number at:

S.E. International, Inc.
P.O. Box 39
156 Drakes Lane
Summertown, TN 38483 USA

Tel. (615) 964-3561
Telex (ITT) 4997468 SOLAR

NOTE: Please do not send us contaminated instruments for repair under any circumstances.

For ANSI standard calibration, contact a certified lab.

LIMITED WARRANTY:

WARRANTOR: S.E. International, Inc., P.O. Box 39, 156 Drakes Lane, Summertown, TN 38483, (615) 964-3561.

ELEMENTS OF WARRANTY: S.E. International, Inc. warrants for 90 days the Geiger Mueller tube and for one year all other materials and craftsmanship in this product to be free from defects with only the limitations or exclusions set out below.

WARRANTY DURATION: This warranty shall terminate and be of no further effect one year (90 days on the GM tube) after the original date of purchase of the product or at the time the product is: a) damaged or not maintained as is reasonable or necessary, b) modified, c) repaired by someone other than the warrantor for a defect or malfunction covered by this Warranty, or d) used in a manner or purpose for which the instrument was not intended or contrary to S.E. International, Inc.'s written instructions. This warranty does not apply to any product subjected to corrosive elements, misuse, abuse, or neglect.

STATEMENT OF REMEDY: In the event that the product does not conform to this warranty at any time while this warranty is effective, the Warrantor will repair the defect and return the instrument to you prepaid, without charge for parts or labor.

NOTE: While the product will be remedied under this warranty without charge, this warranty does not cover or provide for the reimbursement or payment of incidental or consequential damages arising from the use of or the inability to use this product. The liability of the company arising out of the supplying of this instrument, or its use, whether on warranties or otherwise, shall not in any case exceed the cost of correcting defects in the instrument, and after the said one year period (90 days on the GM tube), all such liability shall terminate. Any implied warranty is limited to the duration of this written warranty.

PROCEDURE FOR OBTAINING PERFORMANCE OF WARRANTY: In the event that the product does not conform to this warranty, please write or call us at the address above. S.E. International, Inc. will not accept contaminated instruments for calibration or repair under warranty or otherwise.

NOTE: Before using this instrument, the user must determine the suitability of the product for his or her intended use. The user assumes all risk and liability connected with such use.

SPECIFICATIONS

CALIBRATION: Factory calibration is by pulse generator and is typically plus or minus 10% of full scale relative to Cesium 137. The Monitor 4 is easily calibrated to ANSI standards by a certified lab.

CALIBRATION PROCEDURE: Position the Monitor 4 upright with the back of the instrument facing the source. Adjust the height of the Monitor 4 so that the center of the tube (lengthwise) will be centered with the beam. Measure the appropriate distance from the source to the center of the tube's diameter.

If you need to adjust the calibration, simply remove the two screws from the top of the case and the two screws inside the battery compartment and ease off the back of the case. The trimpot is located above the ON/OFF/AUDIO switch.

SENSOR: LND 712 halogen-quenched uncompensated GM tube with mica end window 1.5-2.0 mg/cm² thick. This tube has 1000 counts per minute per mR/hr for Cesium 137.

ENERGY SENSITIVITY: Detects alpha down to 2.5 MeV; typical detection efficiency at 3.6 MeV is greater than 80%. Detects beta at 50 KeV with typical 35% detection efficiency. Detects beta at 150 KeV with typical 75% detection efficiency. Detects gamma and x-rays down to 10 KeV typical through the end window, 40 KeV minimum through the case.

SATURATION: This instrument has an anti-saturation circuit good to at least 100 times the maximum reading in the highest range.

AUDIO: Built-in piezo electric transducer gives audible indication of each count when switch is in the audio position. Frequency is approximately 3 kHz.

CURRENT DRAIN: Idling current is typically 190 uA.

OPERATING VOLTAGE RANGE: 7-11 volts DC.

VOLTAGE REGULATION: High voltage fully regulated. Less than a 3.0 volt change in output for a 9 volt to 6 volt change in supply voltage.

POWER REQUIREMENT: One 9 volt alkaline battery, NEDA #1604A, or equivalent.

BATTERY LIFE: Up to 2,000 hours at background radiation levels.

TEMPERATURE RANGE: -20°C to +50°C.

WEIGHT: 178 grams (6.3 oz.) without battery.

SIZE: 145mm x 72mm x 38mm (5.7" x 2.8" x 1.5").

ADDITIONAL INFORMATION FOR THE NEWCOMER TO HEALTH PHYSICS

Since our instruments are sometimes purchased by individuals with no background in Health Physics, we thought it would be helpful if we included this addition to our operational manual. Please be sure to read the entire operation manual thoroughly.

MEASURING RADIATION:

The Monitor 4 detects the four main types of ionizing radiation: alpha, beta, gamma, and x-ray. It is calibrated for Cesium 137, but also serves as an excellent indicator of relative intensities for many other sources of ionizing radiation. The level of radiation for gamma and x-rays is measured in milliRoentgens per hour (mR/hr). Alpha and beta radiation are measured in counts per minute (CPM).

The position of the Geiger tube is shown in Illustration 2. The end of the tube has a thin mica end window built into it. This mica window can be broken if struck directly, and it is protected by the screen on the end of the instrument. This alpha window (see Illustration 1) allows alpha particles to reach the mica end window and be detected. The mica end window will also sense low energy beta, gamma, and x-rays that cannot penetrate the plastic case or the side of the tube, which is stainless steel. (Note: Some very low energy radiation cannot be detected through the mica end window. See SPECIFICATIONS for the Geiger tube sensitivities.)

Always approach a source of radiation carefully. Never touch the Monitor 4 to any suspected radioactive substance in order to avoid contaminating the unit itself. Try to make sure you are not walking through or inhaling any radioactive material. Keep in mind that low energy radiation is hard to detect (see BRIEF OVERVIEW OF RADIATION DETECTION below). We recommend that any contact with radioactive materials be avoided whenever possible.

Unless you expect to be measuring high levels of radiation, always keep the range switch in the x1 position. If you are making a measurement and the meter goes off scale to the right, move the range switch to the next higher setting, x10 or x100 if necessary. (Note: The flashes from the count light and the audible beeps are progressively shorter in the x10 and x100 positions.)

Should you detect radioactivity with your Monitor 4, exercise extreme caution to avoid unnecessary exposure and notify the proper authorities. Depending on where you are, this can be your state emergency management agency, the radiation control division of your state health department, or the NRC regional office. If you think you have an emergency on your hands after regular office hours, you can contact your state or local police department for assistance.

If you stand a risk of contamination, try to protect yourself. Although some beta and most gamma radiation can go through protective gear, you can attempt to avoid skin contamination if the radioactivity is airborne. Wear a respirator and some type of overshoes. A full body protective suit may be necessary. You may have to improvise with whatever is on hand. When you leave the radioactive area, remove your protective outerwear and dispose of properly. If you think you have been contaminated, as an additional precaution shower and consult a physician.

We only mention the above procedure in the event you are faced with some type of unavoidable situation. We recommend that contact with radioactive materials and other sources of radiation be avoided whenever possible.

MAKING MEASUREMENTS WITH THE MONITOR 4:

To measure gamma and x-rays, hold the back of the Monitor 4 toward the source of radiation. Low energy gamma and x-rays (10 - 40 KeV) cannot penetrate the side of the Geiger tube, but may be detected through the alpha window.

To detect alpha radiation, position the Monitor 4 so the suspected source of radiation is next to the alpha window. Alpha radiation will not travel far through air so put the source as close as possible (within 1/4 of an inch) to the alpha screen without touching it. Even a very humid day can limit the already short distance an alpha particle can travel. Most alpha radiation can be shielded with a sheet of paper. However, alpha emitters are especially dangerous when inhaled because they may become lodged in the lungs for months or years.

To detect beta radiation, point the alpha window toward the source of radiation. Beta radiation has a longer range through air than alpha particles but can usually be shielded by a few millimeters of aluminum. High energy betas may be monitored through the back of the case.

To determine whether radiation is alpha, beta, or gamma, hold the back of the Monitor 4 toward the specimen (see Illustration 2 for location of the Geiger tube). If there is an indication of radioactivity, it is most likely gamma or high energy beta. Place a piece of aluminum about 1/8" thick between the case and the specimen. If the indication stops, the radiation is most likely beta. (Keep in mind that to some degree most common isotopes contain both beta and gamma.)

If there is no indication through the back of the case to begin with, position the alpha window close to but not touching the specimen. If there is an indication, it is probably alpha or beta. If a sheet of paper will stop the indication it is most likely alpha. (Note: Do not hold the specimen above the alpha window in order to avoid particles falling into the instrument.)

BRIEF OVERVIEW OF RADIATION DETECTION

The Monitor 4 is calibrated for Cesium 137 and is very accurate for it and other isotopes of similar energies. Some of the isotopes that the Monitor 4 will detect relatively well are Cobalt 60, Technetium 99M, Phosphorous 32, Strontium 90, and many forms of Radium, Plutonium, Uranium, and Thorium.

Some forms of radiation are difficult or impossible for our Geiger tube to detect. Some examples of this would be Tritium, radon gas, X-rays from CRT's and TV's, and radioactivity in environmental samples, such as milk, fruit, vegetables, etc. The Monitor 4 does not detect neutron, microwave, RF (radio frequency), lasar, infra-red, or ultra-violet radiation.

On the other hand, the radiation from some isotopes can overexcite a Geiger tube and cause the meter to indicate a higher level of radiation than is actually there. Americium 241 is an example of this phenomenon. Americium 241 is used in some smoke detectors and many different types of industrial density and flow meters.

Although you might have the best intentions, it is possible to draw misleading conclusions from your readings unless you know exactly what you are measuring, which is seldom the case. We tried to design the Monitor 4 to be able to detect the broadest range of ionizing radiation possible and still be in the price range of the average person. It would take a large closet full of very expensive equipment to accurately measure the whole spectrum of ionizing radiation.

Although the Monitor 4 comes with an operation manual geared to people who have no prior knowledge of radiation and/or radiation detection instruments, it does not provide an in-depth understanding of the science of health physics. We encourage you to seek out other sources of information.

There are many books written about radiation, both pro and con. Everyone agrees that radioactive materials can be dangerous. Considering the controversy that surrounds the issue of radiation safety, we suggest that everyone educate themselves as much as possible.

SOME POSSIBLE HOUSEHOLD SOURCES OF RADIATION

SMOKE DETECTORS: Some smoke detectors contain a radioactive isotope as part of the smoke sensing mechanism.

CAMPING LANTERN MANTLES: Some lantern mantles are made with radioactive Thorium. If you must use them, it is suggested that you do not use them in an enclosed area with poor ventilation. Be especially careful not to inhale or ingest the fine powder that is left when they are burned out. Dispose of the ashes in a sealed container so the dust won't be inhaled by someone else.

CLOCKS, WATCHES, AND TIMERS: Many old timepieces have dials and hands highlighted with radium to make them glow in the dark. Tritium is now commonly used to obtain the same effect. Tritium is also radioactive but emits low energy radiation which cannot penetrate the lens of the timepiece. Tritium can only be detected by very sensitive and expensive equipment.

STATIC ELIMINATORS: Static eliminators for reducing static on film and records commonly use polonium, a strong alpha emitter.

JEWELRY: Some gemstones are irradiated to produce more desirable colors. This process can result in residual radioactivity. These gemstones are supposed to be held for a "cooling off" period. However, there have been cases where radioactive gemstones have entered the market.

There have been confirmed reports of some imported cloisonne being glazed with uranium oxide exceeding U.S. limits.

Gold that had been used to encapsulate radium and radon for medical purposes was improperly reprocessed and entered the market as rings and other types of gold jewelry.

ROCK COLLECTIONS: Many natural formations contain radioactive materials. Hobbyists who collect such things should vent the rooms in which these items are stored and be careful to avoid inhaling the fine dust particles which can flake off of these samples.

POTTERY: Certain types of pottery were glazed with uranium oxide. To the best of our knowledge, this practice has been discontinued, although some of these pieces are still in circulation.

GLOSSARY:

ALPHA PARTICLES: Positively charged particles emitted from the nucleus of an atom. Alpha particles are relatively large, and very heavy. Due to its strong charge (+) and large mass, an alpha particle cannot penetrate far into any material. A sheet of paper or an inch of air is usually sufficient to stop most alpha particles. However, the alpha particle's charge produces a high level of ionization along its path, and that can be very damaging to living tissue. It can be very dangerous to ingest or inhale sources of alpha radiation.

BACKGROUND RADIATION: Naturally occurring radiation that is always present. Sources of background radiation include high energy gamma rays from the sun and outer space, alpha, beta, and gamma radiation emitted from radioactive elements in the earth.

BETA PARTICLES: Negatively charged particles emitted from an atom. Beta particles have a mass and charge equal to that of an electron. They are very light particles (about 2,000 times less mass than a proton) and have a charge of -1. Because of their light mass and single charge, beta particles can penetrate more deeply into matter than alpha particles can. A few millimeters of aluminum will stop most beta particles.

CPM (counts per minute): The unit of measurement usually used to measure alpha and beta radiation.

GAMMA RAYS: Short wavelength electromagnetic radiation higher in frequency and energy than visible and ultraviolet light. Gamma rays are emitted from the nucleus of an atom. These high energy photons are much more penetrating than alpha and beta particles.

ION: An atomic particle, atom, or molecule that has acquired an electrical charge, either positive or negative, by gaining or losing electrons.

IONIZATION: The process by which neutral atoms or molecules are divided into pairs of oppositely charged particles known as ions.

IONIZING RADIATION: Radiation capable of producing ionization by breaking up atoms or molecules into charged particles called ions.

RADIATION: The emission and propagation of energy through space or through matter in the form of particles or waves.

RADIOACTIVE: A term commonly used to refer to a source of ionizing radiation.

ROENTGEN (rent-gen): The basic unit of measurement of the ionization produced in the air by gamma or x-rays. One Roentgen (abbreviated R) is the exposure to gamma or x-rays that will produce one electrostatic unit of charge in one cubic centimeter of dry air. One thousand milliRoentgens (1,000m/R) = 1R. This unit is not defined for alpha and beta radiation.

RADIOISOTOPE: A naturally occurring or artificially produced radioactive form of an element.

X-RAYS: Electromagnetic radiation (photons) of higher frequency and energy than visible and ultraviolet light, usually produced by bombarding a metallic target with high speed electrons in a vacuum. X-rays refer to photons emitted by interactions involving orbital electrons rather than atomic nuclei. X-rays and gamma rays have the same basic characteristics. The only difference between them is their source of origin.

APPENDIX H

INTERNAL CHAIN-OF-CUSTODY PROCEDURES FOR COMPUCHEM, ENSECO, WARZYN AND RMT

COMPUCHEM CHAIN OF CUSTODY PROCEDURES

Memo To: Mike McFadden
From: Bob Whitehead, Janet Garrett JB
Date: April 16, 1990
Subject: Warzyn Woodstock Project

In response to Mr. Hurtenbach's comments concerning our SOP for "Internal Chain-of-Custody Procedures", the following changes will be made to the SOP for this project. The SOP will indicate that "...the temperature of the inside of the sample cooler is taken and recorded..." and that "...the pH reading of a sample aliquot is taken with a disposable pH stick to determine whether the sample has been properly preserved. An analytical pH determination, if required, is performed by a qualified laboratory chemist on an aliquot of the sample using a calibrated pH meter...".

Production Planning & Control SOP 1.1: Logging In Samples

The following steps are completed for all samples as they are received by CompuChem Laboratories. (If for any reason a sample requires special handling upon receipt, the Manager of Production Planning and Control is consulted for directions as to the proper handling and documentation of the samples.)

- * Before opening and while inspecting each sample, each employee is required to wear protective clothing (lab coat, safety glasses and gloves). These items need to be worn at all times when in the marked areas (colored line).
- * Inspect each sample container before opening, making sure that it has not been damaged or opened during shipment. For those clients using padlocks, sealing tape, or custody seals, inspect these items to make sure that they are intact and record this observation on the chain-of-custody form (see Example 1, at the end of this SOP). If the custody seals, tapes, or padlocks are broken, contact Customer Service (for commercial samples) or the Sample Management Office (for EPA samples) for permission to continue processing the sample.
- * Each container is opened under the hood and checked for breakage. Check for the condition of the refrigerant (whether any ice remains or whether the cooling packs are solid) and obtain the temperature of a representative sample (liquid samples only) by immersing a clean thermometer in the sample. Record the temperature on the Sample Record (see Example 2, at the end of this SOP).
- * The temperature and pH are recorded on the Log-In-Sheet. See Ex. 3A and 3B lab notices if a variance occurs.
- * Receiving personnel must sign and date all chain-of-custody documentation upon sample receipt and record any discrepancies (sample matrix, for instance) on the chain-of-custody form.
- * The Supervisor of Sample Receiving must verify that the Receiving Clerk has signed and dated the chain-of-custody form.
- * When a CompuChem SampleSaver® is received, the SampleSaver number is recorded on the Log-In-Sheet and is entered into the LMS system by number.

- * Remove samples from the shipping container and compare the sample identification information on the sample bottles to the sample information on the traffic sheets, packing lists, and Chain-of-Custody Form included in the container (see Examples 5A and 5B, at the end of this SOP). If discrepancies exist, note the problem on the chain-of-custody form; the Receiving Clerk notifies Customer Service (for commercial samples) and the Supervisor notifies SMO (for EPA samples).
- * Each water VOA is checked for air bubbles and headspace, and noted on the Chain-of-Custody Form as well as the Log-In-Sheet.
- * On each complete and correct EPA Chain-of-Custody and Traffic Report the statement 'Received in Good Condition' is written or stamped, initialed and dated by the receiving individual.
- * On each complete and correct Commercial Chain-of-Custody the statement 'Received in Good Condition' is written or stamped, initialed and dated by the receiving individual.

'Received in Good Condition' is intended to indicate that the sample or samples were received intact with all associated sample tags (if applicable), custody seals (if applicable), pH for inorganics, and corresponding documentation in order. If there are any discrepancies in the documentation or other problems (such as breakage of the containers or chain-of-custody seals), the exceptions are noted on the appropriate documents, initialed and dated.
- * The statement 'Received in Good Condition' does not, however, include sample temperature since EPA samples are generally received at temperatures above the recommended 4°C. The temperature is noted on the sample Log-In Sheets and the gray envelope.
- * Check incoming samples against SMO scheduled receipts (for EPA samples).
- * Log the sample in on the Log-In-Sheet Log, noting the following items:

Case number	Temperature
CompuChem sample ID	Client name or order number
Receiving date (RD)	Sampling date (SD)
Analysis codes	Matrix
Volume received	pH (Inorganics Samples Only, see PP&C SOP 3.1)

- * For EPA samples, enter the samples' account data into the marketing section of the CLMS in order to generate the order number and associated requisition numbers. For commercial samples, contact customer service to check for the existence of the order and to receive requisitions for analyses. Then complete the order in the CLMS, and complete the EPA Scheduling Log (Example 6, at the end of this SOP).
- * Enter sample into sample receipt portion of CLMS in order to generate a CompuChem number for each sample. Fill in the CompuChem number on the accessioning log sheet (this completes the log sheet).
- * The CompuChem number is a unique, six digit identity which is generated by the LMS system in numerical sequence. It can be cross-referenced to the Client ID.
- * A CompuChem label is generated in numerical sequence, with the CompuChem number.
- * Label samples with the CompuChem number by wrapping each sample bottle with its computer - generated CompuChem sample label. Sample labels are color coded, and are to be rotated with a different color every 2 week period by the Supervisor of Receiving or the Supervisor designee.
- * Each Log-In-Sheet is reviewed by the Supervisor of Environmental Receiving to ensure information is documented. After review each log sheet is stamped as reviewed and initialed and dated.
- * Transfer the labelled samples to the secured, locked walk-in cooler facility.
- * The CompuChem number is listed on the original Chain-of-Custody Form next to the associated client ID when possible.
- * Access the Quiz portion of the CLMS to produce the worksheets for EPA sample analyses. For EPA samples the system will generate volatile, semi-volatile, and pesticide worksheets. For commercial and inorganic samples, pull the appropriate worksheets from the worksheet files; the analysis codes for these samples should have been included with the packing information and confirmed with customer service. All laboratory worksheets are distributed to the appropriate Production Planning and Control Planner.

- * To produce EPA quality control worksheets for the QC samples associated with a batch of samples, access the EPA Water or EPA Solid programs of Quiz in the CLMS and enter the samples' CompuChem Numbers; copy these worksheets on green paper. To assemble commercial QC worksheets, pull the appropriate green fraction worksheets from the trays in the Shipping and Receiving area. Separate QC Sample Records are used to document the analysis of the QC samples associated with a particular batch. These are printed after the entry of sample numbers into the system and are put into green QC folders for Report Integration. Included in the commercial folder are the Sample Record (generated by the CLMS), a copy of the order form, and, if necessary a copy of the Chain-of-Custody Record.
- * Assemble commercial file folders for Report Integration; include in the production sample's folder the Sample Record, Customer Sample Information Sheet and Chain-of-Custody Record; in the green Quality-Control folder include the QC Sample Record, which also goes to Report Integration.
- * Assemble EPA file folders for Report Integration; EPA only has the Sample Record in the file folder. A gray envelope contains all materials for the case including: yellow copy of the OTR (Organic Traffic Report), Chain-Of-Custody, original air-bill, a copy of the Log-In-Sheet, a copy of the EPA scheduling Log, Custody Tags (if received) and a grey envelope contents sheet (See 6b). The white copy of the OTR is returned with a cover sheet to the EPA/SMO (Sample Management Office) (See Attachments 6c and 6d).
- * If problems arise concerning received samples, contact Customer Service (for commercial samples) or the Technical Management Staff (for EPA samples see below):
- * Samples are sometimes received from the EPA that should not have been sent. Therefore, they require a transfer. The following steps should be taken:
 - 1) Fill out a new Chain-of-Custody Form using the information on the sample tags.
 - 2) Sign the Chain-of-Custody in the first section labeled "Relinquished By".

- 3) Copy of Chain-of-Custody is kept for our records; the original is sent with samples. A copy of the paperwork received with the samples should also be sent.
 - 4) Notations made on Traffic Reports stating samples are being sent to another laboratory.
- * Samples hand-delivered after business hours should be recorded as follows:
- 1) Actual date of sample receipt shall be recorded on the Chain-of-Custody.
 - 2) The date and time of sample receipt are recorded on the TRs as follows:

Organic Traffic Report

- a. Date and time of receipt in Column F, sample condition upon receipt.

Inorganic Traffic Report

- a. Same as above.

The notation "H.D." (Hand-Delivered) should be recorded next to the actual sample receipt time and date. The following calendar day shall be recorded in the block designated for Receipt Date on the Organic/Inorganic Traffic Report.

QUALITY ASSURANCE NOTICE

CompuChem# _____
Sample ID _____
Case# _____
Type of Analysis _____
Receipt Date _____

The pH reading for the sample above was _____, the required pH level is _____.

The Client was contacted by a member of CompuChem's Environmental Marketing
Department. The Environmental Receiving Department was instructed to:

Preserve In-House _____
Analyze - Qualify with Notice _____
Dispose - Client will Resample _____
Subcontract Lab to Preserve _____

Supervisor Signature _____
Date _____

QANR2
900130

QUALITY ASSURANCE NOTICE

CompuChem# _____
Sample ID _____
Case# _____
Type of Analysis _____
Receipt Date _____

The required temperature for Environmental samples requiring Organic/Inorganic Analysis is 4C (+/-2C). The temperature on the sample above was _____.

The Client was contacted by a member of CompuChem's Environmental Marketing Department. The Environmental Receiving Department was instructed to:

Analyze - Qualify with Notice _____
Dispose - Client will Resample _____

Supervisor Signature _____
Date _____



№ 000933.

CHAIN-OF-CUSTODY RECORD

[illegible]

Example 2

Sample Record

Requisition Number:
Case:
Turnaround: Priority:
Analysis Codes:

CompuChem Number:
Account Number:
Due:

Lab Instructions

Receiving Data

Sample Identification:
SS Number:
Date Received:
Receiving Information:
Containers Received:
Matrix: Temp:

Time:

SS Code:
Receiving Codes:

Sampling Date(s):

Deliverables

Deliverables Code:

Lab Requirements

Laboratory Completion Date	Repeat Date
----------------------------------	----------------

----- ()

----- ()

----- ()

----- ()

Company Name:

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Example 2 (Cont.)

Sample Record

Requisition Number:
Case:

CompuChem Number:
Account Number:

FOOTNOTES:

Applicable QA Notices:

Company Name:

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COMPUCHEM LABORATORIES											
RECEIVED ON:		ORG/INORG:		ORDER#		# OF PAGES		OF			
CASE #				FREIGHT #'s		CASE ID		TAT			
DELIVERED BY:				REGION:		C-OF-C Y or N		TAGS Y or N			
ACCOUNT #:		CONTRACT #		\$ AMT:							
REMARKS	REQ	SAMPLE ID	CC#	SD	TEMP	Sul	Chl	pH	ANAL. CODES	MX	VOLUME
1											
2											
3											
4											
5											
6											
7											
8											
9											
10											
11											
12											
13											
14											
15											
16											
17											
18											
19											
20											

REVIEWED:

RECEIVED BY:

LOGGED-IN BY:

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COMPUCHEM LABORATORIES										# OF PAGES	OF	
RECEIVED ON:										CASE ID		
CLIENT:										TAT	C-OF-C Y or N	
DELIVERED BY:												
SUBCONTRACT #												
#	REMARKS	RED	SAMPLE ID	CC#	SD	TEMP	Sil	Chl	pH	ANAL CODES	MX	VOLUME
1												
2												
3												
4												
5												
6												
7												
8												
9												
10												
11												
12												
13												
14												
15												
16												
17												
18												
19												
20												

LOGGED-IN BY:

RECEIVED BY:

REVIEWED:

Example 3A

COMPUCHEM LABORATORIES

date shipped _____ date received _____
to consignee: _____ from consignee: _____

number of sample: _____

consignee name: _____

address: _____

DO NOT REMOVE: FOR COMPUCHEM USE ONLY

Example 38

PLEASE NOTE THAT ANY AND ALL PRESERVATIVE(S) ARE TO
BE ADDED BY THE CUSTOMER AT THE TIME OF SAMPLE COLLECTION

At the end of the sampling period, it is vital to ship the sample via express transportation. To insure proper follow-up and prompt analysis, please call 1/800-334-8525 and provide us with the following information:

1. Date Shipped
2. Time Shipped
3. Freight Carrier
4. Freight Bill of Lading Number

Sampling Period

From:

Date _____

Time _____

To:

Date _____

Time _____

Company _____

Address _____

City & State _____ Zip Code _____

Sample Name/Number _____

Return this form in the envelope provided and return with the SAMPLESAVER.

Thank you.

PLEASE NOTE THAT ANY AND ALL PRESERVATIVE(S) ARE TO
BE ADDED BY THE CUSTOMER AT THE TIME OF SAMPLE COLLECTION.

COMPUCHEM LABORATORIES

EPA SCHEDULING LOG

WEEK ENDING SATURDAY:

CASE#: () NEW CASE () OLD CASE () COMPLETED CASE

REGION: TURNAROUND TIME:

ACCOUNT #:

CONTRACT #:

CONTRACT \$

QUANTITY RECEIVED

OF SAMPLES

WATERS
SEDIMENTS/SOILS
OTHER

CONCENTRATION: () LOW () MEDIUM () HIGH

ORDER#:
AIRBILL#:

PROBLEMS/COMMENTS:

SMO CONTACTED AT (TIME)

SPOKE TO:

RESOLUTION FROM SMO:

COMPUCHEM

CONTENTS OF FOLDER

DATE: _____

EPA: () ORGANIC () INORG

COMMERCIAL: ()

CASE #: _____

CLIENT: _____

NO. OF TRAFFIC RPTS: _____

ORDER#: _____

#	ITEM	Y	N	NA	COMMENTS
1	CHAIN-OF-CUSTODY	()	()	()	
2	AIRBILL	()	()	()	
3	DAILY LOG SHEET	()	()	()	
4	TRAFFIC REPORT	()	()	()	
5	TAGS	()	()	()	
6	SMO COVER LETTER	()	()	()	
7	SCHEDULING LOG	()	()	()	

PROBLEMS/COMMENTS:

SAMPLE RECEIVING:

DOCUMENTATION CONTROL:



COMPUCHEM
LABORATORIES

**SAMPLE DELIVERY GROUP (SDG)
TRAFFIC REPORT (TR) COVER 01**

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Lab Name: _____ Contract No.: 68-01-_____

Lab Code: _____ Case No.: _____ SAS No.: _____

Full Sample Analysis Price in Contract: \$ _____

SDG No./First Sample in SDG: _____ Sample Receipt Date: _____
(Lowest EPA Sample Number (MM/DD/YY)
in first shipment of
samples received under SDG)

Last Sample in SDG: _____ Sample Receipt Date: _____
(Highest EPA Sample Number (MM/DD/YY)
in last shipment of
samples received under SDG)

EPA Sample Numbers in the SDG (listed in alphanumeric order):

1 _____	11 _____
2 _____	12 _____
3 _____	13 _____
4 _____	14 _____
5 _____	15 _____
6 _____	16 _____
7 _____	17 _____
8 _____	18 _____
9 _____	19 _____
20 _____	20 _____

Note: There are a maximum of 20 field samples in an SDG.

**Attach Traffic Reports to this form in alphanumeric order
(i.e., the order listed on this form).**

Sample Custodian

Date

ENSECO CHAIN OF CUSTODY PROCEDURES

7. SAMPLE CUSTODY

Upon receipt by Enseco, samples proceed through an orderly processing sequence specifically designed to ensure continuous integrity of both the sample and its documentation.

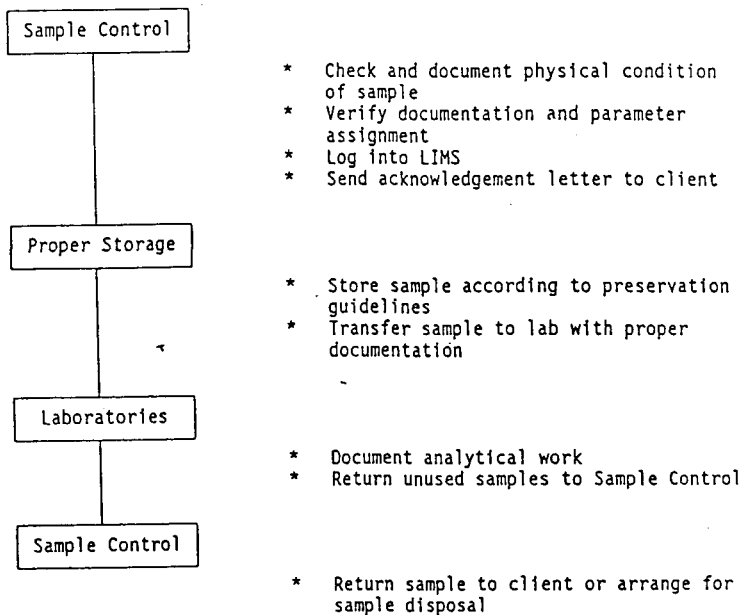
All samples are received by Enseco's Sample Control Group and are carefully checked for label identification, and completed, accurate chain-of-custody records. Photographs document the condition of samples and each sample is then assigned a unique laboratory identification number through a computerized Laboratory Information Management System (LIMS) that stores all identifications and essential information. The LIMS system tracks the sample from storage through the laboratory system until the analytical process is completed and the sample is returned to the custody of the Sample Control Group for disposal. This process is summarized in Figure 7-1. Access to all Enseco laboratories is restricted to prevent any unauthorized contact with samples, extracts, or documentation.

An example of the Enseco Chain-Of-Custody Record used to transmit samples from the client to the laboratory is given in Figure 7-2. The Chain-Of-Custody Record (Interlaboratory Analysis Form) used to transmit samples between laboratories within Enseco is given in Figure 7-3.

Sample bottles provided to the client by Enseco are transmitted under custody using the Enseco "Sample Safe™".

Figure 7-1

ENSECO SAMPLE PROCESSING FLOW CHART



INTERLABORATORY ANALYSIS

SHIP TO: (circle one)	SEND RESULTS TO:
CAL ERCO CLE GAS MAR HOU	Rocky Mountain Analytical Laboratory 4955 Yarrow Street Arvada, CO 80002 (303) 421-6611 FAC: (303) 431-7171
Attention:	Attention:

CLIENT NAME	PROJECT NO.
-------------	-------------

Relinquished by: (Signature)	Received by: (Signature)	Date	Time
------------------------------	--------------------------	------	------

Relinquished by: (Signature)	Received by: (Signature)	Date	Time
------------------------------	--------------------------	------	------

Import Lab ID	Enseco ID	Client ID	Matrix (a, s, w)	Date Sampled	Date Rec'd	Date Auth.	Analysis Requested/ P.L. Item #	Sample Condition Upon Receipt
------------------	-----------	-----------	---------------------	-----------------	---------------	---------------	--	--

Co ID	Client ID	Matrix (a, s, w)	Date Sampled	Date Rec'd	Date Auth.
SAMPLE					

1. Written results required by (date): _____ Verbal results required by (date): _____
- b. QC: ☐ Standard Enesco ☐ CLP Protocol ☐ Project-Specific _____
- c. Sample Disposal: ☐ Enesco ☐ Return to Client ☐ Phone RMAL
- d. Raw Data Copies Needed: ☐ Yes ☐ No
- e. Detection Limits: ☐ Standard Product ☐ Other*
- f. Holding Times: ☐ Enesco ☐ EPA-CLP ☐ Other*
- g. *Special Instructions: _____

RMT CHAIN OF CUSTODY PROCEDURES

STANDARD OPERATING PROCEDURE

PROCEDURE TITLE: Receiving, Log-in, Storage, Analysis and Disposal of Samples

DEPARTMENT: All Departments

PROCEDURE:

Scope

The following steps are taken by the Laboratory Personnel when samples arrive in the laboratory for analysis:

- Case seals and sample seals are examined for integrity.
- Samples are examined for proper labels, damage, proper preservatives and temperature, and referenced to the chain-of-custody.
- The chain-of-custody form (Figure 1.07-1) is signed.
- Samples are stored under the proper environmental conditions, and their location and condition are noted in the appropriate log.
- Samples are entered into the Laboratory LIMS System.
- A Sample Acknowledgment Form is filled out and sent to the project manager.
- Supervisors are notified of the arrival of the samples.
- Internal chain of custody form is completed and samples are stored in a secure area.
- Work lists and work schedules are prepared for sample analysis.
- Both the preparation and analysis of samples will be documented.
- Samples are returned or disposed of after analysis is completed.

Receipt

The company mail clerk, or when unavailable, the sample custodian or his/her designated backup will sign for the receipt of samples from the commercial carrier.

Log-in

Samples should be logged in as soon as possible after receipt. If samples arrive during non-business hours, they must be logged in immediately the next business day. The date of receipt should be the date that the form was completed, and the actual receipt date should be noted in the "comments" section on the custody form.

Before opening each cooler, check for the presence/absence of custody seals or custody tape on each cooler, and record this information in the "Seal # _____ recvd. intact by" box on the RMT Chain-of-Custody form.

Open each cooler and remove the enclosed documents. Note the presence/absence of the Chain-of-Custody, RMT workorder forms, and air bills or bills of lading. These will be noted on the Chain-of-Custody in the "Comments" section or a separate piece of paper if the Chain-of-Custody is absent.

Sign the RMT Chain-of-Custody form in the last box "received by;" note date/time of receipt. Write the air bill tracking number and shipper name on the previous "relinquished by" box providing the air bill is in agreement with the one documented by the shipper. Any discrepancies must be noted. Attach the air bill to the Chain-of-Custody.

Remove all sample containers from the cooler and place them on the log-in counter. Items to check and note condition or presence/absence on the custody form are:

- The condition (intact or broken) of all samples and their respective custody seals, if present.
- The representative temperature of each cooler, using an unpreserved water sample, or in the case of soils or samples with no headspace, the presence of ice sufficient to cool the samples to $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ as per standard RMT procedure.
- The disposal of any damaged samples will be in an appropriate manner and will be documented.
- The pH of each preserved sample and the notation of any preservative added in the designated box on the Chain-of-Custody form.

- Presence/absence of sample tags - For RMT procedures these are usually non-detachable labels affixed to the containers. This should be noted in the "Comments" section of the Custody form.
- Compare the sample tag ID's on the sample to the ID's on the Chain-of-Custody in the space labeled "Sample Station ID."
- RMT workorder forms specifying which analysis is requested on each sample should also accompany the shipment of samples. The presence of this form serves as a check to ensure that the correct bottles for each requested analysis have been received.

LIMS entry

The sample custodian will assign consecutive laboratory sample numbers to each sample received. Laboratory sample numbers will be listed on the Chain-of-Custody to cross reference the sample tag ID numbers. The laboratory sample numbers will be written on each sample bottle on the affixed label or sample tag.

Initiate a Sample Receipt Acknowledgment form after the samples have been entered into the LIMS system. Attach the Sample Receipt Acknowledgment form to the Chain-of Custody form and the work order.

Storage

The location of each type of sample bottle will be noted in the appropriate grid space in the RMT sample log/refrigerator tracking book (Figure 1.07-2).

The storage area will be kept secure at all times. Each sample will be logged into the appropriate refrigerator or bin. The sample custodian will initial and date the separate Refrigerator Tracking Log (Figure 1.07-3) or Bin Log (Figure 1.07-4) sheet kept next to each refrigerator or bin after storing each sample. Each subsequent analyst will sign and date the refrigerator or bin log when removing or replacing samples. All transfers will be documented on an internal chain of custody record (refrigerator or bin log). After a sample has been removed from storage by the analyst, the analyst is responsible for the custody of the sample. Each analyst must return the samples to the storage area before the end of the working day or prior to the end of his/her shift. The refrigerators are maintained according to RMT written procedure.

Samples and extracts will be stored after completion of analysis in the proper storage area. Volatile samples will be stored separately from other samples. Standards will not be stored with samples.

Laboratory Security

Access to the laboratory will be through a monitored area. All laboratory personnel are issued a security card which permits entry to the laboratory. Other outside access doors to the laboratory are kept locked. Visitors will sign a visitors log and be escorted while in the laboratory. Samples will remain in secure sample storage until removed for sample preparation or analysis.

Raw Data

All hand written raw data pertaining either directly or indirectly to a sample analysis must be written in water insoluble black ink. All raw data should be on the appropriate documents (e.g., laboratory notebook, bench sheet, calibration logs, etc.); data must not be recorded on "scratch paper" and then transferred to the appropriate documents. If for any reason data are not recorded on the appropriate documents the paper containing the original data should be, if possible, permanently attached to the document. If this is not possible, the paper should be referenced on the document. Stray marks and numbers on documents are not acceptable; all data must be legible and identified. Columns of data should be labeled, and the units should be defined (i.e., peak area, concentration, absorbance, weight, etc.).

Bench Sheets and Laboratory Notebooks

Any employee collecting raw data must sign and date the data in water insoluble black ink and describe the kind of procedure conducted (e.g., sample extraction, digestion, analysis, etc.). Computer generated data must be signed and dated (if not initialed and dated on the computer printout) by the responsible individual operating that instrument.

It is essential that any unusual occurrences or data that may affect the analysis be recorded and documented in its proper place.

Any spillage, contamination, quality control failure, or other reason that necessitates that a sample be rerun should be documented, and a short explanation should be given.

Unique instrument identification for each instrument used during the collection of raw data should be included on the bench sheet or in the laboratory notebook.

Instruments

All instruments must have an instrument standard operating procedure (SOP), analytical run log, maintenance and calibration log.

Each instrument must be calibrated according to the procedure and the frequency described in the instrument SOP, analytical method or the Quality Assurance (QA) Manual, and the calibration recorded in the log and the samples analyzed. Dates and explanations of routine and non-routine repairs must also be recorded in the maintenance log. Records of non-routine maintenance should document the nature of the defect, how and when the defect was discovered, and any remedial action taken in response to the defect.

Analytical Methods and Procedures

If an analytical method or procedure is modified or if an alternative procedure is used, which is different from the analytical method currently used in the laboratory, the actual method or procedure used should be written on or attached to the raw data. These variations should be justified and approved in writing by the section supervisor. Special requests by clients should be documented in the raw data.

Data Handling and Calculations

All looseleaf data must contain a project number or other identification to designate it as part of a particular project or sample.

All computer calculation sheets must be attached to a bench sheet or stored in an orderly fashion, being cross-referenced in some manner. All manual calculations should be recorded on the bench sheets. If calculation sheets are used, they shall be attached to the bench sheets, or cross-referenced to the bench sheet. All chromatograms and instrument printouts should be labeled with instrument parameters.

Each chromatogram injection should be identified by sample number (or Standard), injection volume, and must be signed and dated by the person responsible for injection. If a sample is auto-

injected, one signature and date are adequate for the group of samples in that run.

Each AA, ICP, Lachat, or other appropriate instrument printout should be identified with sample number (or standard) and must be signed and dated by the person responsible for each analysis. If a sample is auto-injected, one signature and date are adequate for the group of samples in that run.

Standard Curves and Standard Preparation

Record the data for the standard curve on the bench sheet with appropriate units. In those cases where a standard curve is applicable to more than one bench sheet, record on each bench sheet the location of the standard curve data.

Dilutions from stock standards should be labeled with the date the dilution was made and the date that the stock standard was prepared. These dates should be recorded on the bench sheet along with the standard curve data.

Reporting an Assay

It is the analyst's responsibility to review the raw data and the bench sheet prior to being reviewed by a second analyst. The second analyst reviews the data for calculations and data completeness. Upon reviewing the raw data the second analyst signs and dates the bench sheet. The section supervisor reviews the data for quality control (QC) criteria (calibration, frequency of QC samples and QC results). The section supervisor approves the data for LIMS entry by initialing and dating the raw data. The section supervisor reviews the LIMS generated report for technical significance. The report then goes to the laboratory director for final approval. The laboratory director reviews the final report. Upon approval the laboratory director signs and dates the final report. The QC coordinator reviews approximately 10% of the final reports. This is done prior to the review and approval of the report by the laboratory director. The QC coordinator reviews the report for QC criteria, completeness, QA compliance and traceability. Please see the attached flow chart for the process of reviewing raw data in the laboratory.

Sample Disposal

Samples may be completely consumed during analysis, returned to the client or sampling location, stored under required environmental conditions if reanalysis is anticipated or under

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ambient conditions if reanalysis is not likely, or disposed by the laboratory. Samples and extracts shall usually be disposed within sixty days unless otherwise specified.

The Laboratory Director, or his/her designee, shall determine the method and time for disposal if not specified by the Project Manager.

Some waste may be disposed of in a sanitary sewer as permitted by 40 CFR 261.3(a)(2)(iv). Some samples may be hazardous because of their general characteristics or because they are listed in 40 CFR Part 261. shipping of these materials is addressed in 40 CFR 172.02, 172.03, 172.04, 172.300 and 172.400.

[illegible]

LABORATORY REFRIGERATOR TRACKING LOG

[illegible]

WARZYN CHAIN OF CUSTODY PROCEDURES

CHAIN-OF-CUSTODY
Superfund Level

Scope and Application: Chain-of-custody procedures are used to maintain and document sample possession. This particular procedure is applicable when full chain-of-custody procedures are required for enforcement driven investigations.

Reference: NEIC Policies and Procedures, May, 1978 (Revised March, 1986).
EPA-330/9-78-001-R.
U.S. EPA Central Regional Laboratory (10/15/82).

Procedure: Due to the legal nature of enforcement driven investigations, possession of samples must be traceable from the time the samples are collected until introduced as evidence in legal proceedings or destroyed. To maintain and document sample possession, strict chain-of-custody procedures are followed.

A sample is under your custody if:

- 1) it is in your possession, or
- 2) it is in your view, after being in your possession, or
- 3) it was in your possession and you locked it up, or
- 4) it is in a designated secure area.

Field Custody:

1. As few people as possible should handle samples.
2. The field sampler is personally responsible for the care and custody of the samples collected until they are properly transferred or dispatched to the appropriate laboratory.
3. Sample tags and labels shall be completed for each sample, using water proof ink (unless prohibited by weather conditions). For example: a log book notation would explain that a pencil was used to fill out the sample tag/label because a ball point pen would not function in freezing weather.
4. The field project coordinator determines whether proper custody procedures were followed during the field work and decides if additional samples are required.

Transfer of Custody and Shipment

1. Samples will be packaged properly for shipment and dispatched to the appropriate laboratory for analysis, with a separate custody record accompanying each shipment. The method of shipment, courier name, and other pertinent information is entered in the "Remarks" (#14) section of the custody record (see Figure 3). Shipping containers are "sealed" with numbered chain-of-custody seals. The seal numbers are also noted in the "Remarks" section of the custody record.
2. Samples are accompanied by a chain-of-custody record (see Figure 3). When transferring the possession of samples, field personnel will complete the information required for #1 through #11 on the chain-of-custody. Any remarks pertaining to shipping are included in #14. This information is required prior to the field personnel relinquishing custody of the samples. This record documents sample custody transfer from the sampler, to the mobile laboratory or a distant laboratory.
3. Each shipping container will be accompanied by a chain-of-custody record identifying the contents. The original record will accompany the shipment, and a copy will be retained by the field project coordinator.

Sample Receipt and Sample Log-In Procedures

Safety Precautions: All samples received should be considered hazardous and appropriate precautions should be taken when handling these samples. Under no circumstances should any personnel other than the sample custodian or project leader open coolers. If damage or leakage is noted, stay clear of the coolers and notify the sample custodian or project leader immediately. All samples will be opened in a hooded area!

Procedure:

- 1.0 The project leader will notify the sample custodian in writing of incoming samples.
- 2.0 The custodian will receive the samples and deliver them to a hood located in the analytical laboratory. The procedures described below will be followed by the sample custodian:
 - 2.1 Examine the shipping container and record the following information on the project log form (Refer to Figure 1). A separate project log form is required for each shipping container.
 - The presence/absence of custody seal on the shipping container.
 - The condition of the custody seal (i.e., intact, not intact).

- 2.2 Open the shipping container in a hooded area, remove the enclosed sample documents, and record the following information on the project log form.
 - The presence/absence of the chain-of-custody record(s).
 - The presence/absence of airbills and/or bills of lading documenting shipment of the samples.
- 2.3 Remove the samples from the container and record the following information in the project log form.
 - Condition of samples (intact, broken, leaking, cold, etc).
 - The presence/absence of sample tags.
 - Sample tag numbers. Compare these numbers with the chain-of-custody record(s) (Figure 3). If sample tag numbers do not match. Record this fact on both the chain-of-custody record and project log form and contact the data management supervisor so that the discrepancy can be resolved.
- 2.4 Compare the following documents to verify agreement of the information contained on them.
 - Chain-of-custody records.
 - Sample tags.
 - Airbills or bills of lading.
- 2.5 Sign the Chain-of-Custody (Figure 3 - #12 and #13), project log form and airbills.
- 3.0 Login the samples (refer to the "Chain-of-Custody, Log-In and Tracking Procedures" SOP Parts 2-7):
 - 3.1 Each sample is assigned a unique sequential laboratory number. The laboratory number is entered on the chain-of-custody (#15), sample labels and sample tags.
 - 3.2 The sample custodian will remove the sample tags. All tags will be placed in sealed envelopes and placed in the appropriate project file.
- 4.0 Sample Storage:
 - 4.1 Samples and extracts will be stored in a secure area designated for strict chain-of-custody samples.
 - 4.2 Damaged samples will be disposed of in an appropriate manner and the method of disposal documented.

- 4.3 The laboratory and sample storage areas are secure with strict limited access by only laboratory personnel.
 - 4.4 Whenever samples are removed from storage, the removal will be documented. All transfers of samples will be documented on the internal chain-of-custody records. (Refer to Figure 2).
 - 4.5 Samples and extracts will be stored after completion of analysis in accordance with the contract or until sample custodian is instructed otherwise by the enforcement Project Officer.
 - 4.6 The location of stored organic extracts will be recorded.
 - 4.7 VOA samples will be stored separately from organic extracts.
 - 4.8 Standards are stored separate from samples.
 - 4.9 Samples requiring refrigeration are stored in the walk-in coolers. refrigerator. Metal samples are stored in the black cabinets in log-in. Metal digestates are stored on shelves in the metals lab.
- 5.0 Sample Security:
- 5.1 Samples will be stored in a secure area.
 - 5.2 Access to the laboratory will be through a monitored area. Other outside-access doors to the laboratory will be kept locked.
 - 5.3 Visitors will sign a visitors log (located at the reception area and will be escorted while in the laboratory area.
 - 5.4 Refrigerators, freezers, and other sample storage areas are secure as they are located within the laboratory.
 - 5.5 Only laboratory personnel have access to the laboratory.
 - 5.6 Samples will remain in secure sample storage until removed for sample preparation or analysis. All transfers of samples into and out of storage will be documented on an internal chain-of-custody record. An example of an internal chain-of-custody record used for this procedure is attached. (See Figure 2).
 - 5.7 These internal custody records will be maintained in the project file.
 - 5.8 After a sample has been requested from storage by the analyst, the analyst is responsible for the custody of the samples. Each analyst must return the samples to the storage area before the end of the working day.

Internal Chain-of-Custody Procedure:

The following procedure for documentation of internal chain-of-custody for samples requiring continuous custody.

1. Samples requiring refrigeration will be stored in the walk-in coolers located in the sample entry area. Metal samples are stored in the black cabinets in log-in. Metals digestates are located in the metals lab. A list of sample numbers will be maintained by the sample custodian.
2. Only the laboratory personnel will have access to the walk-ins.
3. Samples will remain in the designated walk-ins until removed for sample preparation and/or analysis.
4. All transfer of samples into or out of the walk-ins will be documented on an internal chain-of-custody record (see Figure 2). These records are maintained by the sample custodian.
5. Once a sample is removed from a walk-in by the sample custodian, he/she will then give it to the analyst, who is then responsible for the custody of the sample. Each analyst must return samples to the walk-in before the end of the working day. Samples are not allowed to sit on the bench overnight.
6. The next morning, sample custodian will put samples back in their proper place on the shelves. Sample custodian is responsible for signing sample request sheet that samples were returned properly. Any remarks will also be noted by the sample custodian.
7. When sample analyses and necessary quality assurance checks have been completed by the laboratory or after a 3 month time period whichever is longer, the unused portion of the sample should be disposed of properly. All identifying tags, data sheets, and laboratory records shall be retained as part of the permanent documentation of the project. Tags and forms are never discarded!

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Director, Technical Services

Kim D. Finner
Kim D. Finner
Laboratory Manager

Revision Date

9-26-87

8-18-88

3-16-90

FIGURE 1

Sample Custodian Signature: _____

Date: _____

(Circle Appropriate Response)

Custody See)	present/absent intact/not intact
1	
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Project Number: _____

Chain-of-Custody	present/absent
1. Name of the person who found the evidence	
2. Date and time the evidence was found	
3. Location where the evidence was found	
4. Name of the person who collected the evidence	
5. Date and time the evidence was collected	
6. Name of the person who transported the evidence	
7. Date and time the evidence was transported	
8. Name of the person who stored the evidence	
9. Date and time the evidence was stored	
10. Name of the person who retrieved the evidence	
11. Date and time the evidence was retrieved	
12. Name of the person who presented the evidence	
13. Date and time the evidence was presented	
14. Name of the person who received the evidence	
15. Date and time the evidence was received	

Airbill Number:

Sample Tags	present/absent
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Sample Tags	present/absent
Sample Tag Numbers	listed/not listed on chain-of-custody
1	
2	
3	
4	
5	
6	
7	
8	
9	
10	
11	
12	
13	
14	
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[illegible]

WARZYN SAMPLE REQUEST SHEET

Need Samples By:
Date: _____
Time: _____

I am requesting:

[illegible]

I am returning:

Date/Time:

[illegible]

CHAIN-OF-CUSTODY, LOG-IN AND TRACKING PROCEDURES

Scope and Application: This procedure is applicable to the log-in of samples and addresses such areas as: sample custody, log-in, labeling and preservation of samples. This procedure begins as the samples enter the laboratory and follows the samples to their destruction.

Procedure:

1. Chain-of-Custody Procedure: Samples arriving in the laboratory are accompanied by some type of custody record. The most common type of record is that shown in Figure 1. Follow the instructions below for all levels of chain-of-custody.

A. Superfund Level Chain-of-Custody:

1. Refer to the "Chain-of-Custody - Superfund Level" SOP.

B. Routine Chain-of-Custody:

1. Samples arriving in the laboratory are accompanied by a chain-of-custody record (refer to Figure 1).
2. Review the custody form with the samples received and see that the spaces marked #1 - #12 have been accurately completed by the sampler.
3. Any discrepancies with the chain-of-custody record or the samples should be immediately brought to the attention of the customer service relations person so that the problems can be solved in an efficient manner.
4. If samples were shipped, note the courier name in the remarks section (#15) of the chain-of-custody record.
5. Record any other comments such as: temperature upon receipt, preservation (if done in lab), problems, etc, in the remarks section (#15).
6. Fill in the lab numbers in the section marked #16 (refer to the log-in portion of this procedure for how to obtain lab numbers).
7. Sign and date the chain-of-custody record (#13 - #14).
8. Route the custody record to the data management office when log-in is complete.

NOTE: The laboratory is a secured area with strict limited access. Any samples arriving to the laboratory during "off hours" are stored in the laboratory refrigerator until they are logged in. Laboratory personnel are responsible for the care and custody of the samples during the analysis stage and until the samples are removed from the refrigerator and properly disposed of.

2. Log-In Procedure: Samples are logged in as follows (Refer to Figure 2):

A. Sample Information:

Care should be taken to document the condition of samples upon receipt. Anomalies such as broken or leaking bottles, expired holding times, improper labeling or preservation, and air bubbles in VOA vials should be noted on the chain-of-custody. It should also be noted whether the samples arrived cold.

B. Numbering Samples:

Number the samples on the chain-of-custody form. Record lab numbers used in the black lab book. If no chain-of-custody form accompanied the samples, the sampler must be contacted and one must be filled out. Blank forms are available in the log-in area.

- NOTE:
- Sample preparation is required if the samples need to be filtered and/or preserved in the lab.
 - If metals are requested, metal digestion is required for all matrices except filtered groundwaters or private well.
 - Sample compositing is required if the lab is instructed to composite a series of samples received into one sample for analysis.

C. Subcontracted Work Required:

Refer to the subcontracting SOP for further instructions on subcontracting samples.

D. Special Accounting Notes:

If there is a discount or a special proposal for the sample analyses to be charged, record that information on the chain-of-custody.

E. Labeling Bottles:

1. Record the lab number on the sample bottle label and caps using a water-proof marker.
2. Check that the appropriate preservation and preparation has been circled.
3. If metals and total hardness are both required, using a red water-proof marker, place a dot on the bottle cap of that bottle to be used for those analyses.

F. Preserving Samples:

1. Samples which have been preserved in the field must be checked via pH paper in the laboratory to insure they were preserved to the correct pH. Check preserved samples as follows:
 - a. Acid-preserved (HNO_3 or H_2SO_4) samples must be preserved to a pH <2. Using a pH stick, check the pH. If the pH is not <2, note this on the chain-of-custody, then add additional acid until a pH is obtained. IT IS CRITICAL THAT THE CORRECT TYPE OF ACID BE ADDED.
 - b. Base-preserved NaOH samples must be preserved to a pH > 12. Using a pH stick, check the pH. If the pH is not > 12, note this on the chain-of-custody, then add additional 10N NaOH until a pH > 12 is obtained.
 - c. Base-preserved NaOH with ZnAc samples must be preserved to a pH > 9. Using a pH stick, check the pH. If the pH is not > 9, note this on the chain-of-custody, then add additional 10N NaOH until a pH > 9 is obtained.
2. Samples which have not been preserved in the field need to be preserved in the laboratory. Record any preservation done in the lab on the field sheets or custody records. Refer to Figure 3 for preservation requirements and preserve accordingly. It is important that samples arrive in appropriate containers (see Figure 3). If they do not, contact the inorganic/organic supervisor. They will determine which analyses, if any, can be run.
3. It is important that samples be received and maintained cold for most analyses. If a sample was received warm, make note of this on the field sheet or custody record.

G. Computer Log-In:

Analysis requests are entered into the Laboratory Information Management System (LIMS). The LIMS database is used for scheduling of analytical work and to produce accounting invoices. Analysis requests are entered into the LIMS database as follows:

1. Turn on the computer terminal.
2. Enter: C CHICO (return).
3. Enter: ASCORA (return).
4. Enter: Password (return).
5. At the VMS \$ prompt, enter LABMENU (return).
6. Enter: LOGIN (return).
7. Enter: Password (return).
8. The Log-In screen will appear as shown in Figure 2. Using information on the chain-of-custody, proceed as follows:
 - a. Enter project number (return). Account number and name will be filled in automatically. If the information matches the chain-of-custody, press return.
 - b. Sample date automatically defaults to yesterday's date. If this is correct, press return or over ride with correct date.
 - c. Enter sample identification from station location on the chain-of-custody and press return.
 - d. Enter proper matrix number and press return.
 - e. Desc: This is used if sample identification is longer than the number of characters in the sample identification field. (Return to over ride or enter information and return.)
 - f. Date received automatically defaults to today's date. If correct, press return or over ride with correct date.
 - g. Days to process automatically defaults to 14 days. If this is correct (standard turnaround time) press return or if rush work, over ride with the number of days until due.
 - h. Duedate: First date listed is report date, second date is the lab due date which can be changed if necessary.

- i. Comments: Enter special tests or other information needed to analyze samples correctly and press return.
 - j. Manager: Filled in automatically; press return if correct or over ride.
 - k. Price/Cost: Return if correct or over ride.
 - l. % Markup/Discount: For accounting. Enter a negative number for a discount (i.e. -15 for 15%) and a positive number for markups (i.e. 100 for 2 x price). Check accounting sheets for projects with special pricing.
 - m. To enter parameters, press Next Screen (see Figure 2A).
 - n. Matrix automatically defaults to matrix entered above (return or override).
 - o. Enter the proper code name for the test to be run from report MF01. Use parent codes if applicable for quicker entry. (Press the down arrow key, return.)
 - p. To enter address for the report, press Next Screen (see Figure 2B).
 - q. Type: Report (return).
 - r. If an address has already been entered and matches the chain-of-custody press Commit and return.
 - s. If no address appears, press return until cursor is in the City field. Type in the city and press return.
 - t. Type in state (press Commit and return). After city and state has been entered for the first sample, it will automatically be copied for the rest of the sample numbers in a single log-in.
9. To duplicate the entry for another sample on the same chain-of-custody:
- a. If next sample to be entered has all the same information as the previous sample entered, press Duplicate Record key (F7).
 - b. Enter sample identification and press Commit key, return.
10. To duplicate only the information in the upper block of the log-in screen (sample date, matrix etc.):
- a. Press Create Record key (Insert Here) and enter sample identification.

- b. Press Previous Screen (F12) if sample date needs to be changed.
 - c. Press Next Screen, return. Then press Next Screen again to enter new product codes for the analyses requested.
 - d. Press Previous Screen to enter new sample or to quit.
 - e. Press Clear Form (F17) to enter a new chain-of-custody.
 - f. Press Exit/Cancel (PF4) to exit from the log-in screen.
 - 1. Press Exit/Cancel to exit from the LIMS system.
 - 2. At the VMS \$ prompt, enter LO (return) to log off the VAX computer.
 - 3. Enter N (return).
13. If in Step 8a. the computer says project number is invalid:
- a. Press Exit/Cancel (PF4).
 - b. Enter "Y" (return).
 - c. On the LIMS Main menu, use the up arrow key to position the cursor on "Seedpak Management". Press Commit key.
 - d. Use the down arrow key to move to project entry. Press Commit key.
 - e. Enter project number (return).
 - f. Enter account number (9999) and return.
 - g. Enter description for chain of custody (project name).
 - h. Enter manager from chain-of-custody (i.e. Finner, K).
 - i. Press Exit/Cancel (PF4), twice.
 - j. At the LIMS main menu, select log-in and press Commit.
14. To print daily log-in reports:
- a. Print out the appropriate FS02 reports for only the samples logged in that day. The computer printout must be checked against the custody record or field sheets by the inorganic/organic supervisors so that any errors can be corrected. Print a report as follows:
 - b. Select "Seedpak Reporting" from the main menu, and press Commit.

- c. Select FS02 and press Commit.
 - d. Enter today's date, press Commit and return.
 - e. The inorganic/organic supervisors are responsible for updating and printing lab schedules as they require them.
 - f. Any "rush" work is to be brought to the inorganic/organic supervisors' attention immediately.
15. Sample Storage:
- a. Proper handling and storage is necessary in order to maintain the integrity of the samples. Refer to Figure 3 for storage requirements.
 - b. Refrigerated Storage:
 - 1. Store routine custody samples in the walk-in refrigerator. Separate samples by preservative type. Samples with organic analyte requests are stored on a separate shelf in the walk-in.
 - c. Non-Refrigerated Storage:
 - 1. Metal samples are stored in the log-in area in the appropriate cabinets.
 - 2. Other samples not requiring refrigeration are stored on the appropriate shelves next to the walk-in refrigerator.
 - d. Any samples removed for analysis must be put back in the original location when the analysis is complete.
16. Sample Handling:
- a. Samples are handled in and out of the walk-in coolers and black cabinets by the sample custodian.
 - b. Analysts are responsible for filling out sample request forms (see Figure 4) to request needed samples.
 - c. Forms are given to the sample custodian.
 - d. Sample custodian gathers requested samples and leaves yellow copy of form with samples, and keeps the white copy.
 - e. Analysts return samples to the walk-in accompanied by the yellow copy of the request form.

- f. Next morning, sample custodian puts samples back in their proper place on the shelves.
- g. Sample custodian signs off on white sheet that samples were returned. Any remarks are also noted on the white sheet.

17. Storage/Disposal:

Due to limited sample storage facilities available, the following guidelines have been created for sample storage/disposal. Sample storage time may be extended upon client's request.

- a. One week after the report issue date, inorganic samples which are in cold storage are to be transferred to warm storage.
- b. One month after the report issue date, organic samples which are in cold storage are to be transferred to warm storage.
- c. Samples in warm storage should be disposed of as follows:
 - Aqueous Samples: Dispose 1 month after report issue date.
 - Non-Aqueous Samples: Dispose 6 months after report issue date.

Michael J. Linskens
Michael J. Linskens
Director, Technical Services

Kim D. Finner
Kim D. Finner
Laboratory Manager

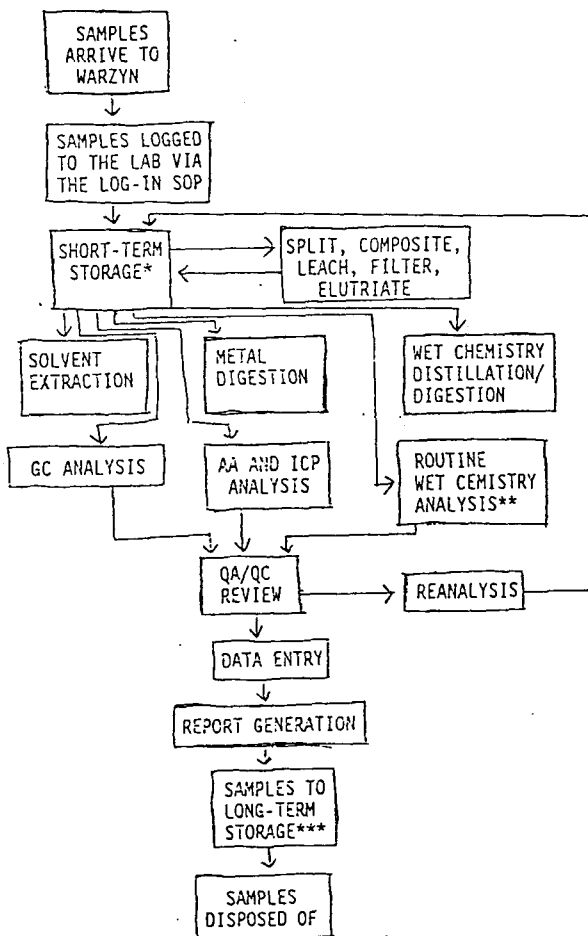
Revision Date

08/18/87

08/15/88

3.18.90

WARZYN ENGINEERING INC.
ANALYTICAL SERVICES
SAMPLE FLOW DIAGRAM



* SHORT TERM STORAGE: WALK-IN REFRIGERATOR FOR SAMPLES REQUIRING REFRIGERATION, BLACK CABINETS IN SAMPLE LOG-IN AREA FOR SAMPLES NOT REQUIRING REFRIGERATION, WALK--INS FOR SAMPLES REQUIRING "SUPERFUND LEVEL CHAIN-OF-CUSTODY".

** ROUTINE WET CHEMISTRY ANALYSES INCLUDE BOTH AUTOMATED AND MANUAL TESTS.

*** LONG TERM STORAGE: NON-REFRIGERATED STORAGE IS LOCATED IN THE FIELD STORAGE ROOM.

FIGURE 1

CHAIN OF CUSTODY RECORD

[illegible]

Login/Order Entry						
Project #: STANDARD		Acct: _____				
Sample #:	Sample Date:	Sample ID: _____				
Matrix:	Desc:	_____				
Received: 14-FEB-90	Days to Process: 14	Due Date: _____				
Comments:	Manager: _____					
Order: _____	on 14-FEB-90	Price/Cost: 0 _____ / 0 _____				
Login: _____	% Markup/Discount: 0 _____					
Matrix	Product	Type	Price	Cost	Hold date	PList
_____	_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____	_____

Project #:		Login/Order Entry	
60818.00		Acct:9999 GPC-576	
Sample #: 496-003		Sample Date: 28-JAN-90 Sample ID: MW-3	
Matrix: 1 GroundH2O		Desc:	
Received: 31-JAN-90		Days to Process: 14 Due Date: 14-FEB-90 12-FEB-90	
Comments:		Manager: CLARK, J	
Order: 619 on 31-JAN-90		Price/Cost: 312 / 0	
Login: 496		% Markup/Discount: 0	

Matrix	Product	Type	Price	Cost	Hold date	PList
1 GroundH2O	AG	S	12	0	12-FEB-90	S
1 GroundH2O	AS	S	25	0	12-FEB-90	S
1 GroundH2O	BA	S	12	0		S
1 GroundH2O	CD	S	12	0	12-FEB-90	S
1 GroundH2O	CR	S	12	0	12-FEB-90	S
1 GroundH2O	DIGASSE	S	0	0		S
1 GroundH2O	HG	S	32	0	25-FEB-90	S
1 GroundH2O	PB	S	22	0	12-FEB-90	S
1 GroundH2O	SDF	S	10	0		S
1 GroundH2O	SE	S	25	0	12-FEB-90	S

v Char Mode: Replace Page 1

Count: 1

Sample Addresses for 496-003

Type #
REPORT 1

Name: _____
 Addr 1: _____
 Addr 2: _____
 City: ROSEVILLE _____
 State/ZIP: MN _____
 Phone: _____

Type

Name: _____
 Addr 1: _____
 Addr 2: _____
 City: _____
 State/ZIP: _____
 Phone: _____

Type

Name: _____
 Addr 1: _____
 Addr 2: _____
 City: _____
 State/ZIP: _____
 Phone: _____

Char Mode: Replace Page 2

Count: *1

FIGURE 3

(Page 1 of 3)

SAMPLE COLLECTION AND PRESERVATIVE REQUIREMENTS

Parameter	Container, (size in mLs)	Preservation	Maximum Holding Time
<u>Wet Chemistry</u>			
Acidity	P.G. (A)	Cool, 4°C	14 days
Alkalinity	P.G. (A)	Cool, 4°C	14 days
NO ₃ s	P.G. (1000)	Cool, 4°C	48 hours
Baron	P. (A)	Cool, 4°C	
Bromide	P.G. (A)	None Required	28 days
Carbon, Total Organic	P.G. (H)	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days
Chemical Oxygen Demand	P.G. (H)	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days
Chloride	P.G. (A)	None Required	28 days
Chlorine, Residual	P.G. (A)	None Required	Analyze Immediately
Cyanide, Amenable	P.G. (1000)	Cool, 4°C, NaOH to pH>12 (1)	14 days (1)
Cyanide, Total	P.G. (1000)	Cool, 4°C, NaOH to pH>12 (1)	14 days (1)
Density/Specific Weight	P.G.	Cool, 4°C	
Fluoride	P.G. (A)	None Required	28 days
Hardness	P.G. (C)	HNO ₃ to pH<2	6 months
Nitrogen, Ammonia	P.G. (H)	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days
Nitrogen, Nitrate	P.G. (H)	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days
Nitrogen, Nitrite	P.G. (A)	Cool, 4°C	48 hours
Nitrogen, Total Kjeldahl	P.G. (B)	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days
Nitrogen, Total Organic	P.G. (B)	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days
Oil and Grease	G. (1000)	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days
pH	P.G. (A)	None Required	Analyze Immediately
Phenolics, Total	G. (500)	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days
Phosphorous, Ortho	P.G. (A)	Filter immediately, Cool 4°C	48 hours
Phosphorous, Total	P.G. (B)	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days
Silica, Dissolved	P. (A)	Cool, 4°C	28 days
Solids, Dissolved	P.G. (H)	Cool, 4°C	7 days
Solids, Settleable	P.G. (1000)	Cool, 4°C	48 hours
Solids, Suspended	P.G. (H)	Cool, 4°C	7 days
Solids, Total	P.G. (H)	Cool, 4°C	7 days
Solids, Volatile	P.G. (H)	Cool, 4°C	7 days
Specific Conductance	P.G. (A)	Cool, 4°C	28 days
Sulfate	P.G. (A)	Cool, 4°C	28 days
Sulfide	P.G. (500)	Cool, 4°C, add 2 mL 2N Zinc acetate/1000 mL plus NaOH to pH9	7 days
Surfactant (MBAS)	P.G. (1000)	Cool, 4°C	48 hours
Thiocyanate	P.G. (500)	Cool, 4°C	
Total Organic Halides (TOX)	Amber Glass (E)	Cool, 4°C	14 days
Turbidity	P.G. (A)	Cool, 4°C	48 hours
<u>Metals</u>			
Hexavalent Chromium	P.G. (A)	Cool, 4°C	24 hours
Mercury	P.G. (C)	HNO ₃ to pH<2	14 days in plastic, 28 days in glass
Metals (all, except above)	P.G. (C)	HNO ₃ to pH<2	6 months
<u>Organics</u>			
<u>Gas Chromatography</u>			
Volatile Organics	VOC vial (2 ea, 40)	Cool, 4°C (J)	14 days (J)
Phenols	Amber glass (H)	Cool, 4°C (K)	7 days (O)
Pesticides and PCBs	Amber glass (H)	Cool, 4°C (L)	7 days (O)
Pesticides	Amber glass (H)	Cool, 4°C (L)	7 days (O)
PCBs	Amber glass (H)	Cool, 4°C	7 days (O)
Polynuclear Aromatics	Amber glass (H)	Cool, 4°C (K), Store in Dark	7 days (O)
Organophosphorus Pesticides	Amber glass (H)	Cool, 4°C, Store in Dark	7 days (O)
Chlorinated Herbicides	Amber glass (H)	Cool, 4°C	7 days (O)
Total Hydrocarbon Screen	Amber glass (H)	Cool, 4°C	7 days (O)
<u>GC/MS</u>			
Priority Pollutants/MSL	VOC vial (2 ea, 40)	Cool, 4°C	
Volatiles	Amber glass (H)	Cool, 4°C	
Semi-volatiles	Amber glass (H)	Cool, 4°C	
Pesticides/PCBs	Amber glass (H)	Cool, 4°C	
<u>CLP Protocol</u>			
Volatiles	VOC vial (2 ea, 40)	Cool, 4°C	
Semi-volatiles	Amber glass (H)	Cool, 4°C	
Pesticides/PCBs	Amber glass (H)	Cool, 4°C	
<u>Radiological Tests</u>			
Alpha, Beta	P.G. (250)	HNO ₃ to pH<2	6 months
Radium	P.G. (1 gal)	HNO ₃ to pH<2	6 months
<u>Bacterial Tests</u>			
Coliform, Fecal and Total	P.G. (F)	Cool, 4°C (F)	6 hours
Fecal Streptococci	P.G. (F)	Cool, 4°C (F)	6 hours
<u>Solids</u>			
BCMA Classification	P.G. (1000) (G)	Cool, 4°C	--
Volatile Organics	VOC vial (140) (H)	Cool, 4°C	--
Semi-volatile Organics	G. (8 oz)	Cool, 4°C	--
Metals	P.G. (8 oz)	Cool, 4°C	--
Inorganics	P.G. (8 oz)	Cool, 4°C	--

Notes

- (A) 1-3 Analyses; 250 mL required
 4-7 Analyses; 500 mL required
 >8 Analyses; 1000 mL required
- (B) 1-3 Analyses; 250 mL required
 4-7 Analyses; 500 mL required
- (C) The metals selenium, arsenic, silver, mercury and hardness require additional volume. Use the following bottle scenario;
 - One 250 mL bottle for all metals excluding above
 - One 250 mL bottle for all metals plus two of the above analytes (except mercury). If mercury is required, an additional 250 mL is required. Label bottle for mercury analyses.
 - One 500 mL bottle for all metals, including all five of the above analytes. Two 250 mL bottles may be used instead.
- (D) Dissolved solids require 100 mL of sample. This volume can be included in the (A) unpreserved bottle.
 The volume required for suspended, total and volatile solids depends on the nature of the sample. Dirty samples require 100-250 mL, clean require 1000 mL. Sample accordingly.
- (E) Collect sample in either a 250 mL amber bottle with inverted cap or 250 or 500 mL amber bottle with teflon septa. Collect with no headspace.
- (F) Collect 250 mL using either sterilized Whirl-Pak bag or nalgene bottle. Collect in Whirl-Pak bags with sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$) additive if the sample is chlorinated.
- (G) Collect the sample in a 1-liter wide-mouth jar. Collect at least 500 grams for analysis.
- (H) Volatile organics may also be collected using two 40-mL VOC vials.
- (I) Add 0.6 g ascorbic acid if residual chlorine is present.
 Maximum holding time is 24 hours when sulfide is present. Optionally all samples may be tested with lead acetate paper before pH adjustments in order to determine if sulfide is present. If sulfide is present, remove by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is then filtered and NaOH is added to pH >12.
- (J) Preserve chlorinated samples with 1 crystal of sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$) per vial.
 Samples can be either preserved with 1:1 HCl or left unpreserved.
 Samples receiving no pH adjustment must be analyzed within 7 days of sampling.
 Samples that contain hydrocarbons (samples from gasoline or fuel oil contaminated sources) must be preserved with 1 drop of 1:1 hydrochloric acid (HCl).
 If either acrolein and acrylonitrile are to be analyzed, the pH should be adjusted to 4-5 with 1:1 hydrochloric acid (HCl). Samples receiving no pH adjustment must be analyzed within 3 days of sampling.

- (K) Add 0.008% sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$) if residual chlorine is present.
- (L) Adjust pH to 5-9 with sulfuric acid (H_2SO_4). The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of Aldrin, add 0.008% sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$) to the sample.
- (M) Collect two (2) 1000 mL amber glass bottles with teflon-lined caps per analysis.
- (N) Collect two (2) 80 oz amber glass bottles with teflon-lined caps for one or both analyses listed. Alternatively, four (4) 1-liter amber bottles with teflon-lined caps may be used.
- (O) The holding time is 7 days until extraction, 40 days after extraction.

BJH/cac
[cac-700-93A]

WARZYN SAMPLE REQUEST SHEET

Requested By: _____
Date/Time Submitted: _____

Need Samples By:
Date: _____
Time: _____

I am requesting:

[illegible]

I am returning:

Date/Time:

[illegible]

APPENDIX I

DOCUMENT CONTROL AND THE EVIDENTIARY FILE

DOCUMENT CONTROL AND THE EVIDENTIARY FILE SYSTEM

ACCOUNTABLE DOCUMENTS

Accountable documents will include all logbooks, field data records, correspondence, sample tags, graphs, chain-of-custody records, and other sample documentation forms used, original data including laboratory bench sheets, photographic prints and planning documents.

FILE STRUCTURE

Documents will be arranged in the evidentiary file using the format attached.

LOGGING OF DOCUMENTS

Documents will be received by the Document Control Officer who will log them and assign a number to each such that documents within each document subclass are separately serialized. An exception to this will be sample tags, chain-of-custody forms or other documents that are numbered prior to assignment for use.

DOCUMENT ACCESS

Project documents will be secured in a separate, locked file cabinet. Access will be limited by the Document Control Officer to project personnel. A check-out log will be maintained as a record of access.

EVIDENCE FILE AUDIT

Upon project completion, the Warzyn Quality Assurance Officer will audit the evidence file for completeness. Results of the audit will be documented on the attached form and kept in the Final Evidence file.

FINAL DISPOSITION OF FILE CONTENTS

The Final Evidence file will be maintained by Warzyn as described in Section XXII, Record Preservation, of the Consent Order. The file will be maintained during the Pendency of the Consent Order and for a minimum of five years after its termination. The PRP shall notify the U.S. EPA in writing within thirty (30) calendar days prior to the destruction of such documents at the end of the five year period.

CAW/caw/PJV

WARZYN

PROJECT SINGLE-FILE STORAGE SYSTEM - FILE CHECKLIST SHEET

(Continued)

<u>CATEGORY</u>	<u>FILE</u>	<u>COMMENTS</u>	<u>REQUIRED CATEGORIES^{1,2}</u>
Laboratory Data	G	Laboratory test data: chemical, geotechnical, materials: original data and quality control data G1-Analytical laboratory data G2-Geotechnical laboratory data G3-Materials testing laboratory data G4-Subcontracted laboratory data	X
Calculations	H	Calculations, quantity estimates, computer printouts of tabularized data; sets of data should be bound separately	X
Photographs/Maps	I	Photographs, stereo pairs, site maps (published), zoning, topography, geology, groundwater, bedrock, negatives	X
Originals	J	Warzyn original reports or drafts	X
Warzyn Reports	K	Copies of the project report or previous pertinent WEI reports	X
Warzyn Drawings	L	Reference list of report drawings; copies or reduced copies of original drawings. Note: original or reduced mylars will be stored separately	X
Other Reports/Drawings	M	Non-Warzyn reports and drawings	X
Miscellaneous	N	Other file information which does not fit into other categories; file must be named.	X
Checkpoint	O	TEMPORARY FILE of checkprints, draft reports or other work in progress. File must be removed upon job completion.	X

1 Main file categories indicated with an X are standard on all projects, but will not be set up by Records until category-specific data is generated.

2 Subcategories may be established by the Project Manager on a project-specific basis (e.g., Superfund projects).

SS/kjw/KEB/DWH
[ckb-400-98]



DOCUMENT AUDIT CHECKLIST*

PROJECT NO. _____

DATE OF AUDIT _____

PROJECT LOCATION _____

SIGNATURE OF AUDITOR _____

FILE LOCATION _____

- Yes__ No__ 1. Have individual files been assembled (field investigation, laboratory, other)?
- Comments: _____
- _____
- _____
- Yes__ No__ 2. Is each file inventoried?
- Comments: _____
- _____
- _____
- Yes__ No__ 3. Is there a list of accountable documents?
- Comments: _____
- _____
- _____
- Yes__ No__ 4. Are all accountable documents present or accounted for?
- Comments: _____
- _____
- _____

* From NEIC Procedure Manual for the Evidence Audit of Enforcement Investigations by Contractor Evidence Audit Teams, EPA-300/9-81-003-R, April, 1984.

- Yes__ No__ 5. Is a document numbering system used?
Comments: _____

- Yes__ No__ 6. Has each document been assigned a document control number?
Comments: _____

- Yes__ No__ 7. Are all documents listed on the inventory accounted for?
Comments: _____

- Yes__ No__ 8. Are there any documents in the file which are not on the inventory?
Comments: _____

- Yes__ No__ 9. Is the file stored in a secure area?
Comments: _____

- Yes__ No__ 10. Are there any project documents which have been declared confidential?
Comments: _____

Yes__ No__

11. Are confidential documents stored in a secure area separate from other project documents?

Comments: _____

Yes__ No__

12. Is access to confidential files restricted?

Comments: _____

Yes__ No__

13. Have confidential documents been marked or stamped "Confidential"?

Comments: _____

Yes__ No__

14. Is confidential information inventoried?

Comments: _____

Yes__ No__

15. Is confidential information numbered for document control?

Comments: _____

Yes__ No__

16. Have any documents been claimed confidential under TSCA?

Comments: _____

APPENDIX J

**INTERNAL FIELD INVESTIGATIONS AND LABORATORY
AUDIT PROCEDURES**

INTERNAL AUDIT PROCEDURES FOR FIELD METHODS

INTERNAL FIELD INVESTIGATIONS AUDIT

This describes a procedure for auditing activities performed during field investigations or remedial actions. The audit addresses the adherence to procedures documented in project plans. These plans may include the project Work Plan, Sampling Plan, Quality Assurance Project Plan and Health and Safety Plan.

WORK ASSIGNMENT

The Warzyn Quality Assurance Manager (QAM) will schedule each audit performed. Audits may be performed by the QAM or by personnel under his/her direction. Audits may be announced or unannounced.

AUDIT PREPARATION

The auditor will review project plans, standard operating procedures, safety plans, or other pertinent project documents for background information. Equipment that may be required for the audit, including safety equipment, will be obtained for use during the audit. The Warzyn Project Manager will be informed that the audit is to take place in order for the auditor to obtain updated information on site conditions.

ON-SITE AUDIT

A briefing is scheduled with the sampling team prior to initiating the audit. The auditor shall briefly describe the audit process and obtain updated information on the field tasks. The audit is the evaluation of adherence to project planning documents and of sample identification and control, chain-of-custody procedures, field documentation, security of evidence and sampling operations. The evaluation is based primarily on the project planning documents.

The auditor will maintain a record of all activities performed during the audit, which may include logbooks, work papers and checklists. The checklists are attached. The auditor must accurately track the dates and times of audit activities and the document numbers that have been reviewed. Included in the record will be the project codes, project location, identification of the

investigators assigned to the project and auditor's name. The checklists must be completed in their entirety and any other pertinent information should be recorded in the "comments" section.

Sample Control

The field audit addresses handling of samples from time of collection through shipping or delivery to the performing laboratory. A sample is typically identified as to the location, date, time and name of person collecting it. A sample tag is used for this purpose. Samples are also typically listed in a Chain-of-Custody record. The auditor should evaluate adherence to sample documentation and control procedures outlined in the project planning documents.

Data from on-site measurements and observations made by field personnel are typically recorded directly into a field logbook or field data record. The auditor should observe this activity or the results.

Sample Tag

Samples are removed from the sample location and transferred to a laboratory or other location for analysis. Before removal, however, each is preserved in accordance with prescribed procedures and the sample is identified with a sample tag. The information recorded in the sample tag typically includes:

- | | |
|------------------|--|
| Project Code | - An assigned number |
| Station Number | - A two-digit number assigned by the Project leader and listed in the project Plan |
| Date | - A six-digit number indicating the year, month and day of collection |
| Time | - A four-digit number indicating the clock time of collection, for example: 0954. |
| Station Location | - The sampling station description, as specified in the project plan |

Samplers	- Each sampler's name is listed
Tag number	- A unique serial number is stamped on each tag
Remarks	- The samplers record of pertinent observations

The sample tag contains an appropriate place for designating the sample as a grab or composite and identifying the type of sample collected for analysis. The sample tags are securely attached to each sample.

After collection, separation, identification and preservation, the sample is typically maintained under chain-of-custody procedures. If the composite or grab sample is to be split, it is aliquoted into similar sample containers. Identical information is recorded on the tag of each split. This identifies the split sample for the appropriate government agency, facility, laboratory or company. In a similar fashion, tags on blank or duplicate samples are marked "Blank" or "Duplicate," respectively, unless otherwise directed.

The auditor will examine a selected number of sample tags for completeness and accuracy. The auditor will determine if the station number and location are identified; the date and time collected are indicated; the type of sample and analysis are specified, the preservative, if used, is identified, and the sampler('s) signature(s) appear on the tag. The auditor will also determine if the station location accurately identifies where the sample was taken and that the sampling methods used were as specified in the project planning documents.

Chain-of-Custody Record

Possession of samples collected during investigations typically must be traceable from the time collected until introduced as evidence in legal proceedings. Chain-of-Custody Records are used for this purpose.

A sample is in your custody if the following criteria are met:

1. It is in your possession; or
2. It is in your view, after being in your possession; or
3. It was in your possession and then locked up to prevent tampering; or
4. It was in your possession and then transferred to a designated secure area.

The auditor will select a predetermined number of Chain-of-Custody records to be audited in the field. The records must be reviewed to determine if the station number and description correspond to the sample tag, if the date and time correspond, if the parameters to be analyzed have been properly identified, and if all custody transfers have been documented and the date and time of transfer recorded.

The auditor will also determine if samples are kept in custody at all times and are handled to prevent tampering. Sampling equipment should also be checked for security and to detect tampering.

Traffic Forms

If samples are analyzed under the Contract Laboratory Program (CLP), Organics and Inorganics Traffic Report forms must also be used. The Organic and Inorganic Traffic Reports are to be executed by the field sampling team and are subject to audit as are the previously discussed documents. This portion of the audit is to observe that the information recorded on the forms is correct and that it coincides with the information on the sample tags and on the Chain-of-Custody Record.

Field Documentation

Observations and measurements during field investigations must be recorded in bound logbooks. These records are intended to provide sufficient data and observations to enable participants to reconstruct events that occurred during the project and to refresh the memory of the investigators, if called upon to give testimony during legal proceedings.

Logbooks: Project logbooks will be reviewed by the auditor during the field investigation audit to see that each is signed and entries are dated. Logbook entries must be legible, written in ink and contain accurate and inclusive documentation of an individual's project activities. Because the logbook forms the basis for reports written later, it must contain only facts and observations. Language should be objective, factual and free of personal feelings or other terminology which might prove inappropriate. Pertinent information should be recorded in these logbooks from the time each individual is assigned to the project until the project is completed. Entries made by individuals other than the person to whom the logbook was assigned must be dated and signed by the individual making the entry.

Photographs: The auditor will review field logbooks to determine if photographs taken are properly documented. When movies, slides or photographs are taken which show sampling sites or provide other documentation, they are numbered to correspond to the logbook entries. The name of the photographer, date, time, site location and site description are entered sequentially in the logbook as photos are taken.

Corrections to Documentation: As previously noted, unless prohibited by weather conditions, original data recorded in logbooks, sample tags, custody records and other data sheet entries are written with waterproof ink. None of the documents listed above are to be destroyed or thrown away, even if they are illegible or contain inaccuracies which require a replacement document.

If an error is made on a document, the individual may make corrections simply by drawing a line through the error and entering the correct information. The erroneous information should not be obliterated.

Sampling Operations

The auditor will review sampling operations to determine if they are performed as stated in project planning documents. The proper number of samples should be collected at the assigned locations. The auditor will check to determine that the samples are in prescribed containers and are preserved in accordance with project plans. The auditor will determine if the required field

measurements and quality assurance checks are performed and documented as directed.

A closing briefing shall be held with the field team leader to verbally review observations. Unresolved problems will be discussed with the Warzyn Project Manager and Quality Assurance Manager.

Reporting

A written audit report is submitted to the Warzyn QAM after completion of the audit. The report shall include copies of checklists. A copy of the report will be given to the Warzyn Project Manager and will become part of the project file.

RCW/bcn/DWH

[bcn-800-70]

FIELD CHECKLIST
Briefing with Project Manager

SIGNATURE OF AUDITOR _____ DATE OF AUDIT _____
PROJECT MANAGER _____ PROJECT NO. _____
PROJECT LOCATION _____
TYPE OF INVESTIGATION _____
(authority, agency) _____

- Yes__ No__ N/A__ 1. Was a project plan prepared? If yes, what items are addressed in the plan?

- Yes__ No__ N/A__ 2. Were additional instructions given to project participants (i.e., changes in project plan)? If yes, describe these changes.

- Yes__ No__ N/A__ 3. Is there a written list of sampling locations and descriptions? If yes, describe where documents are.

- Yes__ No__ N/A__ 4. Is there a map of sampling location? If yes, where is the map?

- Yes__ No__ N/A__ 5. Do the investigators follow a system of accountable documents? If yes, what documents are accountable?

FIELD CHECKLIST
Field Observations

- Yes__ No__ N/A__ 1. Are the number, frequency and types of field measurements and observations taken as specified in the project plan or as directed by the project manager? If yes, where are they recorded?
- _____

- Yes__ No__ N/A__ 2. Are samples collected in the types of containers specified for each type of analysis? If no, what kind of sample containers were used?
- _____

- Yes__ No__ N/A__ 3. Are samples preserved as required? If no or N/A, explain.
- _____

- Yes__ No__ N/A__ 4. Are the number, frequency and types of samples collected as specified in the project plan or as directed by the project manager? If no, explain why not.
- _____

- Yes__ No__ N/A__ 5. Are samples packed for preservation when required (i.e., packed in ice, etc.)? If no or N/A, explain why.
- _____

- Yes__ No__ N/A__ 6. Is sample custody maintained? How?
- _____

FIELD CHECKLIST
Document Control

- Yes__ No__ N/A__ 1. Have unused and voided accountable documents been returned to the coordinator by the team members?

- Yes__ No__ N/A__ 2. Were any accountable documents lost or destroyed? If yes, have document numbers of lost or destroyed accountable documents been recorded and where are they recorded?

- Yes__ No__ N/A__ 3. Are samples identified with sample tags? If no, how are samples identified?

- Yes__ No__ N/A__ 4. Are sample tags completed (e.g., station no., location, date, time, analyses, signatures or samples, type, preservatives, etc.)? If yes, describe types of information recorded.

- Yes__ No__ N/A__ 5. Are samples collected listed on a chain-of-custody record? If yes, describe the type of chain-of-custody record used and what information is recorded.

- Yes__ No__ N/A__ 6. If used, are the sample tag numbers recorded on the chain-of-custody documents?

- Yes__ No__ N/A__ 7. Does information on sample tags and Chain-of-Custody Records match?
- _____
- _____
- _____
- Yes__ No__ N/A__ 8. Does the Chain-of-Custody Record indicate the method of sample shipment?
- _____
- _____
- _____
- Yes__ No__ N/A__ 9. Is the Chain-of-Custody Record included with the samples in the shipping container?
- _____
- _____
- _____
- Yes__ No__ N/A__ 10. If used, do the sample traffic reports agree with the sample tags?
- _____
- _____
- _____
- Yes__ No__ N/A__ 11. If used, are blank samples identified?
- _____
- _____
- _____
- Yes__ No__ N/A__ 12. If collected, are duplicate samples identified on sample tags and Chain-of-Custody Records?
- _____
- _____
- _____
- Yes__ No__ N/A__ 13. If used, are spiked samples identified?
- _____
- _____
- _____
- Yes__ No__ N/A__ 14. Are logbooks signed by the individual who checked out the logbook from the project coordinator?
- _____
- _____
- _____

- Yes__ No__ N/A__ 15. Are logbooks dated upon receipt from the project coordinator?
- _____

- Yes__ No__ N/A__ 16. Are logbooks project-specific (by logbook or by page)?
- _____

- Yes__ No__ N/A__ 17. Are logbook entries dated and identified by author?
- _____

- Yes__ No__ N/A__ 18. Are photographs documented in logbooks (e.g., time, date, description of subject, photographer, etc.)?
- _____

- Yes__ No__ N/A__ 19. If film from a self-developing camera is used, are photos matched with logbook documentation?
- _____

- Yes__ No__ N/A__ 20. Are sample tag numbers recorded? If yes, described where they are recorded.
- _____

- Yes__ No__ N/A__ 21. Are calibration of pH meters, conductivity meters, etc., documented? If yes, describe where this is documented.
- _____

- Yes__ No__ N/A__ 22. Are amendments to the project plan documented? If yes, describe where the amendments are documented.
- _____

FIELD CHECKLIST
Debriefing with Project Manager

Yes__ No__ N/A__

1. Was a debriefing held with project manager and/or other participants?

Yes__ No__ N/A__

2. Were any recommendations made to the project participants during the debriefing? If yes, list recommendations?

COMPUCHEM INTERNAL LABORATORY AUDIT PROCEDURE

10.0 Corrective Action

10.1 Introduction

Generally, there are two types of corrective actions that may be required when data quality falls below specified limits. The first type, and the simplest to implement and document, is corrective action required because routine data quality assessments are out-of-control. Surrogate and spike standard recoveries, relative percent differences between duplicates, internal standard response variations, and unacceptable blank contamination are some of these assessments in the first category. These are all performed on a sample-by-sample and/or batch basis, and corrective action is limited to evaluating the data with respect to SOP criteria, and accepting or rejecting the sample/batch. The decision that is made is clearly indicated on analytical worksheets, and unless a trend is observed during the course of data validation, additional corrective action or documentation is not necessary.

The second type of corrective action is that required when other, more global QC/QA assessments, are made. The assessments might typically indicate systematic deficiencies or those affecting data useability for more than one batch (i.e., glassware contamination checks, standards preparation errors, etc.). In most cases, assessments of this nature are made by reviewing peripheral QC/QA documentation, observing procedures for comparison with SOPs or GLPs, or receiving feedback from data reviewers, management or those external to the organization (clients, auditors).

The following sections describe the QA reporting and feedback channels designed to ensure that early and effective corrective action is taken in such instances.

In many cases, depending on the nature of the deficiency and the urgency for remedial action, a Corrective Action Report (following this section) will be completed. The report serves to document the deficiency, the required corrective action, and accountability for the action.

For observations made over longer periods of time, the QA Department issues formal summary reports to management on a monthly or quarterly basis. Following is a brief discussion of the types of reports issued to management to assess the overall effectiveness of the QA Program and to reinforce the application of Good Laboratory Practices (GLPs).

CORRECTIVE ACTION REPORT

DATE: _____

PROBLEM / DEFICIENCY:

IDENTIFIED BY: _____

REFERRED TO: _____ (QA)

CORRECTIVE ACTION TO BE TAKEN:

TARGET DATE: _____

FOLLOW-UP AND/FI FINDINGS:

RESOLVED? _____ DATE: _____

SOP REQUIRED TO BE WRITTEN/MODIFIED? YES [] NO [] TARGET DATE: _____

This form to be filed with the Quality Assurance Clerk for permanent record.

10.2 Routine QC Check Reports

The following routine quality control checks (also discussed in section 9.2 of the QA Plan) are performed to verify that samples are not contaminated during transportation, preparation, analysis or storage, and that standards prepared internally are traceable to certified sources.

- Vendor-Supplied Glassware Checks
- Glassware Decontamination Checks
- Water Purification Systems Checks
- Glassware Storage Cabinet Checks
- Refrigerated Storage Systems Checks
- Reagent Purity Checks
- Standards Preparation and Traceability Checks

The criteria for these QC checks and corrective action steps are detailed in the QA SOP Manual. Results are tabulated and/or plotted on control charts, and records reviewed by the QA staff. A series of quarterly reports to management summarize this information and the status of these programs.

10.3 Monthly QA Activity Reports

These reports are produced by all members of the QA staff, and summarize key QA activities during the previous month. The reports are distributed to the Director of QA, and are provided as an attachment and referenced in the Director's report to the CEO, the Executive Staff and senior laboratory management.

Included in these reports is a summary of significant quality problems observed during the period, and the corrective actions taken to remove deficiencies. The report stresses proactive measures that are being taken to improve quality or ensure compliance with QA program requirements.

Laboratory management uses the report to quantitatively measure monthly performance in terms of the number of samples processed, the frequency of repeated sample analyses due to unacceptable QC performance, and the cause of the unacceptable performance. These data are all presented in tables, Pareto control charts or attribute control charts, based on the characterization of each analysis in the Computerized Laboratory Management System (CLMS) using a system of analytical "condition codes."

The Condition Code System is used to monitor sources of data failures. Condition code definitions are provided in an SOP to data generators and reviewers who are responsible for assigning the appropriate code to each analysis (see Appendix D). Each two-letter code is used to characterize the cause of a sample failure or the final status of the data package prior to release to the client.

Various computer programs may be used to sort condition code data according to sample matrix and method. This system is used to pinpoint sources of error, provide feedback to management, reinforce good laboratory practices, and document laboratory performance over time. The QA staff also note in the Monthly QA Activities Report any corrective actions taken or necessary procedural changes, based on the application of condition codes.

Other items included in this report are:

- Summary of any changes in certification/accreditation status
- Involvements in resolution of quality issues with clients or agencies
- QA organizational changes
- Notice of the distribution of revised documents controlled by the QA Department (i.e., SOPs, QA Plan)
- Training and safety issues, if not already covered in audit reports during the period
- Performance of subcontractor laboratories (also communicated in separate, detailed subcontractor audit report to management)
- Positive feedback for acceptable performance on interlaboratory or intralaboratory tests or successful completion of audits.

10.4 Laboratory Performance Reports

This quarterly report presents a statistical and graphical summary of the laboratory's performance on batch-associated quality control samples analyzed over the period. Included are tables, Shewhart control charts and I-charts (for individual data points) for all surrogate and spike standard recoveries. Additionally, a monthly report to the Director of QA presents control charts and tables for all Laboratory Control Sample (Blank Spike) and Blank recoveries. The charts and tables are used primarily to document historical performance, update recovery control limits, and monitor long-range trends that might not be apparent to data reviewers evaluating data on a sample/batch basis.

10.5 Laboratory Audit Reports

Quarterly audit reports are written by a member of the QA staff and distributed to management, and summarize the results of internal laboratory Performance Audits, Systems Audits and Security/Access Audits. When external auditors are involved in Performance or System Audits, a report is written within the next week by the QA staff member coordinating the audit. The report, summarizing audit results as discussed in the debriefing as well as other observations, is distributed to the CEO and senior lab management. The report includes corrective actions required as a result of the audit, and a schedule for implementation. A follow-up audit, usually within three weeks of the distribution of this report, is conducted to verify that corrective actions have been implemented.

Performance Audits

Performance Audits are checks made by a QA staff member or other independent auditors to evaluate the quality of the data produced by the analytical system. These audits are performed independent of and in addition to routine quality control checks, and reflect as closely as possible lab performance under normal operating conditions.

These audits involve the review of approximately 10% of all analytical data reports generated by the lab for calculation and data validation procedures, and overall data quality. Errors observed during the audit are characterized as "critical" or "correctable" and tabulated. If necessary, based on audit findings, an amended data report may be sent to the customer. Following this section is a copy of the QA Audit Summary used by auditors to tabulate the data

for summary into the Quarterly Performance Audit report. A thorough discussion of these audits is included in the QA SOPs. The reports are used by laboratory managers to provide feedback to staff members and establish goals for improved performance.

A number in interlaboratory and intralaboratory tests are conducted routinely at CompuChem[®], and the results are included in individual Performance Audit reports specific to each test. When new methods are available to the laboratory or new personnel are being trained, Laboratory Proficiency Tests are performed. These tests consist of quadruplicate blank spikes, containing a full complement of tests parameters to be analyzed by the method. The replicate results are analyzed by a QA staff member, who generates a summary report to the Director of QA. This report includes the standard deviation and mean recovery for each of the replicate parameters, and the data are used to statistically validate method and/or personnel proficiency. For a thorough discussion of the method validation procedures used, refer to Appendix A of the QA Plan.

On a quarterly basis, blind intralaboratory check samples are introduced into the system by the QA Department. Parameters and methods are chosen for these studies based upon independent (interlaboratory) tests from certifying agencies (including the U.S. EPA and various state agencies), Laboratory Proficiency Test results, Method Validation studies, or results from routine batch-related QC samples. The existence of these check samples in the system is known only to those personnel involved in preparing the samples and scheduling the analytical requirements into the CLMS. A thorough report, detailing the entire data generation and support functions, is completed by the QA staff and reviewed by

the Director of QA before distribution to the CEO and senior laboratory management.

CompuChem² also participates in a number of external, interlaboratory performance studies. These are required as part of various agencies' certification/accreditation programs. As a member of the USEPA's Contract Laboratory Program (CLP), the laboratory is required to successfully analyze quarterly, blind proficiency samples for both organic and inorganic parameters. The CLP program also requires an annual on-site inspection by principals from the USEPA (and their contracted agents). These audits generally follow the same format described below, Systems Audits.

CompuChem² also participates in a number of state certification programs, including those for North Carolina, New Jersey, New York and Florida. All of these programs require the laboratory to submit to annual on-site inspections in order to maintain certification to perform testing on samples originating in the state. All states also require successful performance on inter-laboratory check samples, submitted at least annually, though some reciprocity with the two NC programs (one for drinking water and one for wastewater certification) and USEPA-CLP is allowed under certain circumstances.

Several states utilize the laboratory's performance on the annual Water Supply (WS) and Water Pollution (WP) proficiency testing series, originating out of the EPA Environmental Monitoring and Support Laboratory's performance on all interlaboratory and intralaboratory check samples, tabulated by parameter and method, so negative performance trends can be readily pinpointed.

System Audits

A System Audit is an on-site inspection and review of the QA Program for the total laboratory. While Performance Audits are a quantitative appraisal, System Audits are for the most part qualitative in nature. The System Audit may be either scheduled or unannounced before it is conducted, but occurs routinely on at least a quarterly basis. The auditor reviews the laboratories' SOPs to verify compliance with procedures and activities actually in place. Personnel and facilities are also evaluated during the System Audit. The auditor is required to investigate anything which seems in conflict with the QA Plan, the laboratory or QA SOPs, or Good Laboratory Practices.

If deficiencies are observed during a Performance Audit, and if deemed necessary, the QA Department initiates a System Audit. The audit emphasizes the actions necessary to correct deficiencies noted in the Performance Audit. A Corrective Action Report is completed, detailing all remedial actions taken, and reviewed by the Director of QA. The report must indicate the proposed implementation date and the individual(s) responsible for the action.

Many of the objectives of a routine System Audit are similar to those a client or independent auditor would hope to accomplish during an On-Site Laboratory Evaluation and Data Audit. These goals include ensuring the following:

1. The quality control, including necessary corrective actions, are being applied
2. Adequate facilities and equipment are available to perform the client's required scope-of-work
3. The personnel are qualified to perform the assigned tasks
4. Complete documentation is available, including sample chain-of-custody

WARZYN INTERNAL LABORATORY AUDIT PROCEDURE



INTERNAL AUDITS

Scope:

Internal audits are performed on a quarterly basis. The auditor reviews each area of the laboratory using the attached check list. The quality assurance specialists are responsible for auditing and they report to the director of Technical Services. The auditor prepares a final audit report summarizing their findings and forwards the report to the laboratory manager. The laboratory manager informs upper management of the contents of the audit report, and outline plans to address recommendations or deficiencies.

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LABORATORY CUSTODY AND DOCUMENTATION CHECKLIST

SIGNATURE OF AUDITOR _____ DATE OF AUDIT _____

LABORATORY _____

LABORATORY LOCATION _____

PROJECT IN EFFECT _____

1. Name of Sample Custodian and other personnel responsible for sample receipt and document control.

2. Where are the Sample Custodian's procedures and responsibilities documented?

3. Where are written Standard Operating Procedures (SOPs) pertaining to receipt of samples documented (laboratory manual, written instructions, etc.)?

4. Where is the receipt of Chain-of-Custody Record(s) with samples being documented?

5. Review sample receipt documentation to assure that the nonreceipt of Chain-of-Custody record(s) with samples being documented.

6. Where is the integrity of the shipping container(s) being documented (custody seal(s) intact, container locked or sealed properly, etc.)?

7. Review the sample receipt documentation to assure that the lack of integrity of the shipping container(s) is being documented (i.e., evidence of tampering, custody seals broken or damaged, locks unlocked or missing, etc.)?

8. Determine by asking the Sample Custodian or reviewing the laboratory SOP manual, if agreement among forms, Chain-of-Custody records, and sample tags is being verified? State source of information.

9. Where is the agreement or nonagreement verification (#8) being documented?

10. Review sample receipt documentation to assure that sample tag numbers are recorded by the Sample Custodian?

11. Where are written Standard Operating Procedures (SOPs) pertaining to sample storage documented (laboratory manual, written instructions, etc.)?

- 12a. Do written SOPs and actual laboratory practices demonstrate laboratory security?

- 12b. Describe sample storage area (upright refrigerator in GC lab, walk-in cooler in sample receiving area, etc.).

13. How is sample identification maintained?

14. How is sample extract (or inorganics concentrate) identification maintained?

15. How are samples that require preservation stored to maintain their preservation?

16. Are written Standard Operating Procedures (SOPs) pertaining to sample handling and tracking documented?

17. What laboratory records are used to record personnel receiving and transferring samples in the laboratory?

18. Affirm that each instrument used for sample analysis (GC, GC/MS, AA, etc.) has an instrument log? List those instruments which do not.

19. Determine where analytical methods are documented and ask if methods are available to the analysts?

20. Determine where quality assurance procedures are documented and ask if procedures are available to the analysts?

21. How are written Standard Operating Procedures (SOPs) for compiling and maintaining sample document files documented?

22. How are sample documents filed (by project number, internal laboratory number, batch number, sample number, etc.)?

23. Review sample document files to determine if a document file inventory is prepared for each project file.

24. Review sample document files to determine if all documents in the case files are consecutively numbered according to the file inventories.

25. Observe the document file storage area to determine if the laboratory document files are stored in a secure area.

26. Has the laboratory received any confidential documents?

Complete questions 27, 28 and 29 ONLY if the response to question 26 was yes.

27. Review the project files to assure that confidential documents are segregated from other laboratory documents.

28. Review the project files to assure that confidential documents are stored in a secure manner.

29. Review recommendations from the previous audit to determine if the recommendations have been implemented. If not, the recommendations should be repeated and the laboratory director and the Project Officer should be notified.
-
-

PROJECT DOCUMENT AUDIT CHECKLIST

PROJECT NO. _____

DATE OF AUDIT _____

PROJECT LOCATION _____

SIGNATURE OF AUDITOR _____

FILE LOCATION _____

Yes__ No__

1. Have individual files been assembled (field investigation, laboratory, other)?

Comments: _____

Yes__ No__

2. Is each file inventoried?

Comments: _____

Yes__ No__

3. Is there a list of accountable documents?

Comments: _____

Yes__ No__

4. Are all accountable documents present or accounted for?

Comments: _____

Yes__ No__

5. Is a document numbering system used?

Comments: _____

Yes__ No__

6. Has each document been assigned a document control number?

Comments: _____

Yes__ No__

7. Are all documents listed on the inventory accounted for?

Comments: _____

Yes__ No__

8. Are there any documents in the file which are not on the inventory?

Comments: _____

Yes__ No__

9. Is the file stored in a secure area?

Comments: _____

Yes__ No__

10. Are there any project documents which have been declared confidential?

Comments: _____

Yes__ No__

11. Are confidential documents stored in a secure area separate from other project documents?

Comments: _____

Yes__ No__

12. Is access to confidential files restricted?

Comments: _____

Yes__ No__

13. Have confidential documents been marked or stamped "Confidential"?

Comments: _____

Yes__ No__

14. Is confidential information inventoried?

Comments: _____

Yes__ No__

15. Is confidential information numbered for document control?

Comments: _____

Yes__ No__

16. Have any documents been claimed confidential under TSCA?

Comments: _____

LABORATORY CHECKLIST

Yes__ No__

1. Is there a laboratory procedures manual?

Comments: _____

Yes__ No__

2. Is there a laboratory quality control procedure?

Comments: _____

Yes__ No__

3. Is there a person who reviews that QC and QA in the lab (i.e., QC Officer)?

Comments: _____

Yes__ No__

4. Is there a procedure for the development and review of laboratory procedures?

Comments: _____

Yes__ No__

5. Are procedures updated/reviewed at a set interval?

Comments: _____

Yes__ No__

6. Is the procedure status log current?

Comments: _____

Yes__ No__

7. Is there documentation that each analyst has read and understood each procedure that is applicable to their job requirements?

Comments: _____

Yes__ No__

8. Is there a documentation system that records the analyst's proficiency in the manipulation of laboratory equipment and techniques required in analyses, and that he is knowledgeable and skilled in performing the analyses for which he is responsible?

Comments: _____

Yes__ No__

What is the system?

Comments: _____

Yes__ No__

9. Is there a written training procedure for new analysts?

Comments: _____

Yes__ No__

10. Does the laboratory have QC charts for each parameter for each type of control sample?

- a. Duplicate
- b. Split
- c. Spiked
- d. Preservative blanks

Comments: _____

Yes__ No__

11. Are the QC chart limits $\pm 2SD$ for warning limits; $x \pm 3SD$ for control limits?

Comments: _____

Yes__ No__

12. Are replicate analyses (7) run annually for all parameters?

- a. Precision
- b. Accuracy

Comments: _____

Yes__ No__

13. Are charts current?

Comments: _____

Yes__ No__

14. The percent of laboratory resources devoted to QC and QA is:

- a. 0 - 5%
- b. 5 - 10%
- c. 10 - 20%
- d. >20%

Comments: _____

Yes__ No__

15. Does the laboratory participate in any "round robin" split and/or performance sample programs? ...
Note: List and indicate results.

Comments: _____

Yes__ No__

16. Are externally prepared performance standards obtained from the EPA analyzed yearly for each parameter?
List.

Comments: _____

Yes__ No__

- 17A. Is there a written procedure for cleaning sampling equipment and containers?

Comments: _____

Yes__ No__

- 17B. Does this procedure include the collection of blank samples from collection equipment to assure/document that equipment will not contaminate samples?

Comments: _____

Yes__ No__

- 17C. Are blank samples collected routinely from cleaned sample containers to assure/document efficiency of cleaning?

Comments: _____

Yes__ No__

18. Is there a procedure for data reporting?

Comments: _____

Yes__ No__

19. Do the final data reports indicate if there were variations in the parameter-specific holding times?

Comments: _____

Yes__ No__

20. Is there a written procedure for:

- a. Significant figures?
- b. Rounding off?
- c. Calculation rounding?

Comments: _____

Yes__ No__

21. Are data and laboratory records kept for a specified length of time (i.e., NPDES: 3 years)?

Comments: _____

Yes__ No__

22. Are log books:

- a. bound?
- b. pages numbered?
- c. dated and signed?
- d. reviewed?

Comments: _____

Yes__ No__

23. Are laboratory notebooks:

- a. properly stored?
- b. properly labelled?
- c. complete/accurate?

Comments: _____

Yes__ No__

24. Are the field data logbooks complete and current?

Comments: _____

Yes__ No__

25. Are the receiving logbooks complete and current?

Comments: _____

Yes__ No__

26. Are calibration reagents of unimpeachable purity and product quality as required by each analysis?

Sources List (NBS)

- a. weights
- b. certified thermometers
- c. filter, etc.

Comments: _____

Yes__ No__

27. Is there a procedure to assure that reagents and chemicals Reagent blanks, method blanks checked 100% against specification quality?

Comments: _____

Yes__ No__

28. Are NBS standard reference N/A to most environmental analyses materials used as a routine part of calibration and QC program?

Comments: _____

Yes__ No__

29. Are chemicals properly stored to assure quality?

Comments: _____

Yes__ No__

30. Are there written requirements for all analytical instruments for:

- a. daily warmup?
- b. standardization?
- c. calibration?
- d. optimization procedures
- e. maintenance?
- f. documentation (logs)?
- g. replacement, cleaning, checks, adjustment by laboratory staff and/or service personnel?

Yes__ No__

31A. Is there a written calibration procedure for all measuring and test equipment?

B. Does this procedure specify use criteria?

C. Are calibration standard, reagents, and accessory equipment listed?

D. Does the procedure specify the documentation used in maintenance logbook?

Comments: _____

Yes__ No__

32. Are all instruments tagged with date of last calibration, calibrator, and due date for next calibration?

Comments: _____

ENSECO INTERNAL LABORATORY AUDIT PROCEDURE

12. PERFORMANCE AND SYSTEM AUDITS

Enseco laboratories participate in a variety of federal and state certification programs, (including the U.S. EPA CLP), that subject each of the laboratories to stringent system and performance audits on a regular basis. A system audit is a review of laboratory operations conducted to verify that the laboratory has the necessary facilities, equipment, staff and procedures in place to generate acceptable data. A performance audit verifies the ability of the laboratory to correctly identify and quantitate compounds in blind check samples submitted by the auditing agency. The purpose of these audits is to identify those laboratories that are capable of generating scientifically sound data. Enseco is certified to perform environmental analyses under programs administered by the U.S. EPA, U.S. Army, U.S. Navy, and over 15 states. The most current list of Enseco certifications is available upon request.

In addition to external audits conducted by certifying agencies or clients, Enseco regularly conducts the following internal audits:

- Quarterly systems audits conducted by the Divisional QA Director.
- Periodic (at least yearly) audits conducted by the Corporate QA Office.
- Special audits by the Divisional QA Director or Corporate QA Office when a problem is suspected.

Enseco laboratories also routinely analyze check samples as described below:

- Laboratory Control Samples (DCS, SCS, and method blanks) are analyzed at a frequency equal to at least 10% of the total number of samples analyzed (see Section 11).
 - All Enseco laboratories participate in the analyses of EPA check samples provided under the Water Supply (WS) and Water Pollution (WP) Performance Evaluation Studies. The results of these PE samples are tabulated by the Corporate QA Office to identify performance trends within the Enseco laboratories.
-

-
- The majority of the Enseco laboratories are CLP labs and thus analyze organic and/or inorganic CLP PE samples on a quarterly basis. The results of these analyses are also tabulated and evaluated by the Corporate QA Office.
 - The laboratories participate in multiple state certification programs (including New York, New Jersey and California) which require that PE samples be analyzed periodically.
 - Blind check samples from an independent commercial firm are sent to the laboratories periodically by the Corporate QA Office. The frequency and type of samples sent is based on problem areas identified by evaluation of tabulated PE results.

The results of these check samples are used to identify areas where additional training is needed or clarification of procedures is required.

15. CORRECTIVE ACTION

When errors, deficiencies, or out-of-control situations exist, the QA program provides systematic procedures, called "corrective actions," to resolve problems and restore proper functioning to the analytical system.

Laboratory personnel are alerted that corrective actions may be necessary if:

- QC data are outside the acceptable windows for precision and accuracy;
- Blanks, DCS or SCS contain contaminants above acceptable levels;
- Undesirable trends are detected in spike recoveries or RPD between duplicates;
- There are unusual changes in detection limits;
- Deficiencies are detected by the QA department during internal or external audits or from the results of performance evaluation samples; or
- Inquiries concerning data quality are received from clients.

Corrective action procedures are often handled at the bench level by the analyst, who reviews the preparation or extraction procedure for possible errors, checks the instrument calibration, spike and calibration mixes, instrument sensitivity, and so on. If the problem persists or cannot be identified, the matter is referred to the laboratory supervisor, manager and/or QA department for further investigation. Once resolved, full documentation of the corrective action procedure is filed with the QA department. Corrective action documentation is routinely reviewed by the VP of QA.

RMT INTERNAL LABORATORY AUDIT PROCEDURE

14.0 QUALITY ASSURANCE/QUALITY CONTROL AUDITS

Audits of the RMT Laboratory are conducted for several reasons:

- . To identify potential or actual deficiencies so that the problems may be corrected.
- . To assure that RMT procedures and methods are being followed.
- . To determine that records are properly filled out and filed.
- . To assure that regulatory requirements are met.
- . To establish that quality assurance objectives are met.

Audits are not conducted to assign blame.

14.1 LABORATORY AUDITS

Monthly, the QC Coordinator, shall conduct an in-depth audit of the RMT Laboratory. This audit shall include the following items:

- . Sample maintenance
 - Are stated temperatures for sample storage provided?
 - Are samples processed and tested within prescribed holding times?
 - Are samples properly logged in?
- . Calibration
 - Are calibrations performed as required?
 - Are they properly documented in instrument log books, or as part of project data if required?
 - Do calibration results indicate a trend in instrument performance?
- . Preventive maintenance
 - Are adequate spare parts available?
 - Do specific instruments have repeated maintenance problems?
 - Is preventive maintenance performed and properly documented?

- . Receipt and storage of standards, chemicals, and gases
 - Are all reagents, chemicals, and gases purchased for use in the laboratory of adequate grade for the intended use?
 - Are certifications of material compositions provided when required?
 - Are materials adequately stored to prevent degradation?
 - Are materials kept beyond stated shelf life?
 - Are internal standards properly prepared and stored?
 - Are internal standards kept beyond stated shelf life?
- . Analytical Methods
 - Are the methods used appropriate for project requirements?
 - Are alternate methods approved for use?
- . Data Verification
 - Are data processed and validated as prescribed?
- . Records Management
 - Are the records of analyses complete and properly identified?
 - Are documents submitted to the record system in a timely manner and are they properly maintained?

The following areas shall be audited:

- . Sample Entry
- . Inorganic Laboratory
- . Organic Laboratory
- . Report Production

The laboratory audit shall consist of a general audit and a specific method/procedure audit.

- . A general audit shall be an overview of the whole laboratory from sample receipt to sample disposal for compliance with the Quality Assurance Manual (Figure 14-1).
- . A specific method/procedure audit shall be a detailed in-depth review of an actual method or procedure. This may include

sample receipt (Figure 14-2), standard/reagent/solution preparation (Figure 14-3), sample preparation/extraction (Figure 14-4), sample analysis (Figure 14-5) or data verification (Figure 14-6).

After the general and/or specific audits have been conducted, the Quality Assurance Coordinator shall complete the Laboratory Audit Record form (Figure 14-7). Any problems, observations, and findings which are identified by the Quality Assurance Coordinator shall be discussed with the Group Supervisors.

A written report (Figure 14-8) summarizing the findings of the audit shall be sent to the Group Supervisors who shall be responsible for corrective action. The Group Supervisor, or the employee designated to respond to the report, shall sign and date the report.

The original copy of the audit report form with responses shall be routed to the Laboratory Director for review and comments. The Laboratory Director shall sign and date all audit response forms to document that they have been reviewed. The audit reports forms shall be sent to the Quality Assurance Coordinator for filing.

Deficiencies reported as a result of participation in round-robin studies or outside audits shall be handled in the same manner.

APPENDIX K

DATA VALIDATION PROCEDURE FOR EVALUATION OF INORGANIC DATA

DATA VALIDATION PROCEDURE FOR EVALUATING INORGANIC DATA

Scope and Application:

This standard operating procedure (SOP) describes Warzyn's procedure for evaluating inorganic data according to procedures specified in a Quality Assurance Project Plan (QAPP). Each analytical procedure is specific to a project and is found or referenced in the QAPP. The reviewer must evaluate whether the procedure was adhered to and that the required Quality Control (QC) requirements were met as described in the QAPP and evaluate the usability of the data.

References:

1. Laboratory Data Validation Functional Guidelines for Evaluating Inorganics Analyses, Hazardous Site Evaluation Division, U.S. EPA, July, 1988 (or most current).
2. Statement of Work for Inorganics Analysis, U.S. EPA Contract Laboratory Program, SOW No. 788, July, 1988 (or most current).
3. Quality Assurance Project Plan (QAPP) containing the analytical procedures required for evaluation. Each QAPP is specific to a project.

Requirements:

1. It is recommended that the reviewer be familiar with the RAS Inorganics validation process (refer to the Validation Guidelines and Statement of Work above) prior to evaluating this data. The evaluation and action criteria described in this SOP are very similar to the RAS Inorganics validation process.
2. All completed validated data packages must include the following:
 - a. Data Validation Narrative
 - b. Validated Results; with data qualifiers added and pages stamped "VALIDATED".

NOTE: It is not necessary to include the raw data with the completed validated data package, since a copy of the raw data package already exists in the project file. Only include raw data if there are major concerns or deficiencies with the data set.

Procedure:

1. Screen the data package to see that the samples and analytes undergoing evaluation concur with what was requested on the "Request for Data Validation" form (refer to Figure 1). If discrepancies occur, contact the data management coordinator for clarification prior to continuing.

2. Review the QAPP and the specific analytical procedure for the analyte undergoing evaluation. They will contain:
 - a. A summary of the method required and method reference,
 - b. The applicable matrices and recommended hold times,
 - c. An approximate number of samples and matrix,
 - d. Special technical instructions,
 - e. Required deliverables,
 - f. Data Requirements (Detection Limits, Precision, etc.), and
 - g. Quality Control Requirements.
3. The reviewer must evaluate the data according to the quality control criteria specified in the QAPP. The evaluation of each analyte, in most cases, will include a review of the following criteria:
 - a. Methodology,
 - b. Raw Data/Quality Control Deliverables,
 - c. Detection Limits,
 - d. Holding Times,
 - e. Calibration (Initial and Continuing),
 - f. Blanks (Calibration and Preparation),
 - g. Laboratory Control Samples (EPA Reference Samples),
 - h. Laboratory Duplicates,
 - i. Matrix Spikes,
 - j. Sample Result Verification,
 - k. Field Duplicates,
 - l. Other (as specified in the procedure),
 - m. Overall Assessment for a Case.

4. Evaluate the data for the criteria above as follows:

A. METHODOLOGY

Evaluation

1. Examine the data to determine if the acceptable analytical methodology was used as indicated in the QAPP.
2. Determine if the analytical procedure was followed.

Action

1. If an acceptable analytical method was not used, contact the Project Manager. It is the Project Manager's responsibility to inform the reviewer to continue the validation process of the data as received, or to reject the data. In some cases reanalysis or

resampling may be required, however the decision is left up to the Project Manager. The Project Manager may require that the reviewer determine the "usability" of the data package as submitted prior to deciding what formal action should be taken.

2. If the analytical procedure was not followed as stated, use professional judgement in determining whether the data should be qualified as estimated or unusable.

B. RAW DATA/QUALITY CONTROL DELIVERABLES

Evaluation

1. The QAPP will specify what information should be submitted as part of the data package. Determine whether the data package submitted is complete as described in the QAPP. Usually the following will be required:
 - Method used,
 - Calibration/standardization information,
 - Bench records tabulating the order of analysis of samples, standards, blanks, duplicates and spikes with the resulting instrument readouts and final concentrations. Records submitted should be complete enough such that all results could be recalculated from this raw data.
 - Photocopy of all instrument readouts (strip charts, printer tapes, etc.)
 - Reference (QC) samples to be identified by source, lot number and sample number, with corresponding true values and 95% confidence limits provided.
 - Custody records

Action

1. If the data package is not complete, contact the data management coordinator. The data management coordinator will contact the appropriate laboratory or project manager and obtain the information necessary to complete the evaluation process.

C. DETECTION LIMITS

Evaluation

1. Evaluate whether the detection limits required in the QAPP were achieved by the method used.

Action

1. If detection limits were not reported at least to what was required in the QAPP, review the raw data to determine if the detection limits were achievable. If they were, recalculate the data and report results to the required detection limits.
2. If detection limits were not achievable by the laboratory, determine why (laboratory sensitivity problem or detection limits unobtainable for the method used. Inform the project manager of

the situation. The project manager will determine whether the evaluation should be completed.

D. HOLDING TIMES

Evaluation

1. Determine whether the established holding times were met. The holding time is established by comparing the DATE SAMPLED with the DATE OF ANALYSIS found on the raw data. Required hold times should be noted in the QAPP.
2. Examine the digestion/distillation logs to determine if samples were preserved at the proper pH.

Action

1. If hold times were not noted in the QAPP, use the EPA-recommended hold times and apply these hold times to both water and soil samples.
2. If holding times and preservation criteria are not met, qualify all results > Instrument Detection Limit (IDL) and estimated (J) and results < IDL as estimated (UJ).
3. Use professional judgement in cases where the holding time is grossly exceeded. The expected bias would be low and the reviewer may determine that results < IDL are unusable (R).

E. CALIBRATION

Evaluation

1. Verify that the instrument was calibrated daily and each time the instrument was set up using the correct number of standards and blank. In cases where a curve set-up is not applicable (i.e. a titrimetric procedure), verify that the titrants were standardized as required in the procedure.
2. Verify that the correlation coefficient is ≥ 0.995 (unless otherwise specified).
3. Verify that the calibration verification standards (ICV/CCV) were run at the appropriate frequency and that results were within acceptable limits as stated in the QAPP.
4. Recalculate approximately 10% of the ICV/CCV using the following equation:

$$\%R = \frac{\text{Found}}{\text{True}} \times 100$$

Due to possible rounding discrepancies, allow results to fall within 1% of the contract windows.

Action

1. If the minimum number of standards as required in the QAPP were not used for initial calibration, or if the instrument was not calibrated daily and each time the instrument was set up, qualify the data as unusable (R).
2. If the correlation coefficient is < 0.995 (or as specified in the QAPP), qualify results $> IDL$ as estimated (J) and results $> IDL$ as estimated (UJ).
3. If standardization of titrants was required and not performed, use professional judgement in qualifying the data. The reviewer should review ICV/CCV, EPA reference sample results and other criteria prior to determining if the data should be qualified as estimated (J if $> IDL$; UJ if $< IDL$) or unusable (R).
4. If the ICV/CCV %R falls outside the acceptable limits, use professional judgement to qualify associated samples. If possible, indicate the bias in the review. The following guidelines may assist the reviewer in qualifying the data:
 - If the ICV/CCV %R is significantly greater than the upper control limit (UCL), qualify associated results $> IDL$ as unusable (R); results $< IDL$ are acceptable.
 - If the ICV/CCV %R is significantly lower than the lower control limit (LCL), qualify all associated data as unusable (R)
 - If the ICV/CCV %R are outside the acceptable limits, yet relatively close to the limits, qualify associated results as estimated (J if $> IDL$; UJ if $< IDL$ and the %R is below the LCL).

F. BLANKS

Evaluation

1. Review the raw data for all blanks and verify the results were accurately reported.
2. No contaminants should be found in the blanks (i.e. all blank results should be $< IDL$).
3. Positive blank results must not be corrected by subtracting off the blank value from the sample results.

Action

1. Sample results $> IDL$ but less than 5 times the maximum concentration found in any blank should be qualified as not detected (U).
2. Any blank with a negative result whose absolute value is $> IDL$ must be carefully evaluated to determine if the sample data is biased in any way.

G. LABORATORY CONTROL SAMPLES (EPA REFERENCE SAMPLES)

Evaluation

1. The laboratory control sample (LCS) serves as a monitor of the overall performance of the analysis including all preparation steps. Review the LCS and verify that the results fall within the control limits required. (If no limits noted, use 80-120% for the control limits.)
2. Check the raw data to verify the reported recoveries. Recalculate one or more recoveries (%R) using the following equation:

$$\text{LCS \%R} = \frac{\text{LCS Found}}{\text{LCS True}} \times 100$$

Action

1. If results are < IDL and the LCS recovery is above the UCL, the data are acceptable.
2. If the LCS recovery for any analyte falls within the range of 50-LCL, or > UCL, qualify results > IDL as estimated (J).
3. If results are < IDL and the LCS recovery falls within the range of 50-LCL, qualify the affected results as estimated (UJ).
4. If the LCS recovery results are < 50%, qualify the data for the affected analytes as unusable (R).
5. If a LCS was required and not run, use professional judgement to determine whether the data is estimated (J) or unusable (R).

H. LABORATORY DUPLICATES

Evaluation

1. Review the duplicate data and verify that the results fall within the criteria required. If no criteria exist, use the limits of 20% for the Relative Percent Difference (RPD) or \pm IDL if results are < 5x IDL (35 RPD or \pm 2x IDL if > 5x IDL for soils).
2. Review the data and verify that the duplicate analysis was not performed on a field blank.
3. Check the raw data and recalculate one or more RPD using the following:

$$RPD = \frac{|S-D|}{(S+D)/2} \times 100$$

where, S = Original sample value
D = Duplicate sample value.

Action

1. If duplicate analysis results for a particular analyte fall outside the appropriate control windows, qualify the results for that analyte in all associated samples of the same sample matrix as estimated (J).
2. If the field blank was used for duplicate analysis, carefully evaluate all other quality control data and use professional judgement in evaluating the data.

I. MATRIX SPIKES

Evaluation

1. Review the matrix spike data and verify that the results fall within the limits specified. If no criteria exists, use the limits of 75-125% for both water and soil matrices.
2. Check the raw data and recalculate the % Recovery of at least 10% of the data. Use the following equation to verify that the results were calculated correctly:

$$\%R = \frac{(SSR - SR)}{SA} \times 100$$

Where, SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Added

3. Review the data and verify that the field blank was not used for the spike analysis.

Action

1. If the spike recovery is > 125% (or > UCL stated in the procedure) and the sample results are < IDL, the data is acceptable for use.
2. If the spike recovery is > 125% or < 75% (> UCL or < LCL stated in the procedure) and the sample results are > IDL, qualify the data for these samples as estimated (J).
3. If the spike recovery falls within the range of 30-74% (30-LCL) and the sample results are < IDL, qualify the data for these samples as estimated (UJ).
4. If any spike recovery results are < 30% and the sample results are < IDL, qualify the data for these samples as unusable (R).

5. In cases where more than one spike analysis was performed and one or more %R results were outside acceptable ranges, use professional judgement when qualifying the data. Matrix spikes should be performed on each sample matrix. Qualify data of similar matrix as the spiked sample if different matrices in the data package.
6. If the field blank was used for the spike analysis, carefully evaluate all other quality control criteria and use professional judgement in evaluating the data.

J. SAMPLE RESULT VERIFICATION

Evaluation

1. Examine the raw data to verify the analyte quantitation was calculated as stated in the procedure. Re-calculate a minimum of 10% of the sample results to verify results were calculated correctly.
2. Examine the raw data for anomalies such as baseline drift, negative absorbances (indicative of interferences), omissions, etc.
3. Verify there are no transcription or reduction errors (dilutions, percent solids, sample weights) on at least 10% of the samples.
4. Verify that all results fall within the calibrated range.

Action

1. If any discrepancies are found and can be corrected, make the corrections and note. Increase the level of quality control if necessary. If the discrepancy cannot be resolved by the reviewer, contact the data management coordinator. The laboratory or appropriate agency will be contacted and the information necessary to complete the evaluation will be requested. The project manager should be informed of the delay in the validation process. If any discrepancy remains unresolved, use professional judgement in qualifying the data.

K. FIELD DUPLICATES

Evaluation

1. Field duplicate samples measure field and lab precision and therefore the results may have more variability than lab duplicates. Review the field duplicate data and calculate the RPD. No criteria exists for evaluating the data.

Action

1. If the RPD is $> 50\%$, note in the narrative. Use professional judgement in qualifying any data. Review the raw data to verify that no reduction errors exist. This is to verify that the discrepancy is due to sampling techniques (sampling, preservation,

filtering, etc.) rather than laboratory error. Make notation of the discrepancy in the narrative.

L. OTHER (as specified in the procedure)

Evaluation

1. Review the procedure for any other quality control criteria not covered under the above sections. Examples of additional information required may be: ICP interference check samples, special AA quality control checks, distilled/digested blanks and standards, standardization, specific methodology requests for different concentration levels, etc..

Action

1. If specific quality control criteria were required and not followed, the project manager should be informed.
2. If the data was outside the acceptable ranges, use professional judgement in qualifying the data. Clearly identify your action and justification of the action in the narrative.

M. OVERALL ASSESSMENT FOR A CASE

1. It is appropriate for the reviewer to make professional judgements and express concerns and comments on the validity of the overall data package. This is particularly appropriate when several QC criteria are outside specifications. It is the reviewers responsibility to thoroughly document and explain all data validation qualifiers added to the data.
2. The following is a summary of data validation qualifier definitions which can be used in evaluating the data:
 - U The material was analyzed for, but was not detected above the level of the associated value. The associated value is either the sample quantitation limit or the sample detection limit.
 - J The associated value is an estimated quantity due to quality control criteria not being met.
 - R The data are unusable. The analyte may or may not be present.
 - UJ The material was analyzed for, but was not detected. The associated value is an estimate and may be inaccurate or imprecise.
5. Once the data has been evaluated against all of the points described in part 4, the evaluator can prepare the data validation narrative.

The data validation narrative should be a summary of the data quality indicating any problems or deficiencies found with the data set. If

possible, on data qualified as estimated, note if the data is biased high or low. It is easiest to discuss each analyte separately. Refer to Figure 2 for the form to be used when writing the data validation narrative.

6. Record any data validation qualifiers (DVQ) to the left of the results on the analytical report. The laboratory qualifiers (LQ) will usually be recorded to the right of the result by the laboratory who performed the analytical work. A copy of the lab qualifier definitions should be supplied with the data. Data entry people need to be able to distinguish between DVQ and LQ, so note location of qualifiers if located other than above.
7. Record the sample ID and date sampled as noted on the Chain of Custody on the analytical report.
8. Highlight POSITIVE-HIT data with a highlighter.
9. Stamp all of the analytical reports "VALIDATED". A "VALIDATED" stamp can be obtained from the data management coordinator.
10. Sign and date the Data Validation Request Form.
11. A quality control review needs to be performed on the validated data package. The entire validated data package (including raw data, evaluator's notes and narrative) should be submitted to the QC reviewer for a final review.

Quality Control Review:

1. The purpose of this quality control review is to verify that the data were validated according to the guidelines above, that all data validation qualifiers were added and also that the narrative is complete.
2. Review the validated data package for completeness. The data package should contain the following:
 - Data Validation Request Form
 - Inorganic Data Validation Narrative
 - Validated Analytical Results
3. Verify that results exist for all samples and all analytes requested in the data package.
4. Review the data validation narrative with the validated results for consistency, i.e., if the narrative stated that a particular analyte was qualified, verify that the qualifier has been recorded with the associated data. Check approximately 10% of the qualifiers. If discrepancies are found, increase the level of QC checks.

5. Review the narrative; check that the data has been evaluated according to the quality control criteria specified in the QAPP.
6. Verify that all positive-hit data have been highlighted and that the sample descriptions and sampling dates have been recorded on every result page.
7. Verify that the reviewer has signed and dated the validated data package.
8. If no discrepancies exist, sign and date the Data Validation Request Form. If the data package requires clarification, review the raw data and/or obtain clarification from the reviewer.
9. Deliver the final validated data package to the Data Management Coordinator when the quality control review is completed.
10. Recycle any raw data. Raw data is not submitted with the final validated data package.

APPROVED BY:

Kevin J. Domack
Environmental Scientists Section Leader

Daniel W. Hall
Environmental Division Director

REVISION DATE:

INORGANICS
DATA VALIDATION NARRATIVE

Site Name _____ Project # _____

SMO Case # (SAS #) _____ #Samples/Matrix _____

Laboratory _____ Hours for Review _____

SOW # _____ EPA Validation Guidelines # _____

Sample Numbers _____

Validated By _____ Date _____

Reviewed By _____ Date _____

SUMMARY OF REVIEW:

CAW/d1k/KJD
[d1k-601-22]
60721-MD

APPENDIX L

WARZYN'S DAILY INSTRUMENT QC CHECK PROCEDURE

INORGANIC - DAILY QUALITY CONTROL PROCEDURE

Scope and Application: Daily check of dessicants, instruments, refrigerators and freezer, deionized and Milli-Q water systems to ensure data produced each day is of top quality.

Dessicant: Dri-Rite, an anhydrous compound, is kept in the bottom of each dessicator. When Dri-Rite is blue, it is ready to use, but when a pink shade begins to form, it must be dried in an oven set at 103-105°C until no pink color can be detected. Record date dried in comments section. Record initials and date of daily check in the log book.

Refrigerators and Freezer: Temperature of each of the refrigerators are to be recorded in the equipment record book daily.

<u>Instrument</u>	<u>Acceptable Range</u>
Walk-in cooler #1	4.0°C \pm 2.5°C
Walk-in cooler #5	4.0°C \pm 2.5°C
Silver Raetone #2	4.0°C \pm 2.5°C
Gold General Electric #3	4.0°C \pm 2.5°C
White Frigidaire #4	4.0°C \pm 2.5°C
BOD Incubator	20.0°C \pm 1.0°C

Notes:

1. Each of the refrigerators, freezers and incubators should routinely be defrosted when ice build-up occurs.
2. If fluctuations in temperature do occur, report the occurrence to the lab supervisor immediately. Note in maintenance records.

Instruments: In-lab balances, pH meter, and conductivity meter are to be checked daily against known standards. An Instrument Operating Procedure (IOP) is located in each of the instruments representative log book.

Balance: The Sartorius and Cahn balances are calibrated with standard weights kept in a dessicator. The weights are to be handled with plastic forceps. Each weight is to be recorded in its representative place in the log book. Acceptable ranges are listed in each IOP.

pH Meter: The pH meters are calibrated with fresh buffers daily according to the pH SOP. Record the reading of the pH 10.00 buffer in the log book also. The acceptable range for the pH 10.00 buffer is 9.90 - 10.10.

Note: The inner electrode solution should be checked periodically to ensure accurate readings.

Conductivity Meter: The Lab-Line or the YSI conductivity meter is checked daily against a KCL standard of known concentration. Check the meter according to the IOP located in the log book. The acceptable range is 90-110% of the true value.

Deionized and Milli-Q Water Systems:

D.I.: The D.I. system functions with ion-exchange cartridges located in the sample storage room. Two warning lights are attached to the tanks to ensure they are operating properly. Each day, these lights must be checked. If one or both are out, the supplier must be notified to replace the tanks. Record tank change dates in the log book.

Note: If both lights are out, D.I. water cannot be used until tanks are replaced.

Milli-Q: The Milli-Q water should be checked with a conductivity meter. Conductivity should be no greater than 1 umhos/cm @25°C. The Milli-Q system calibration must also be checked daily. This is done by turning on system and letting pressure build, then depress test button. Reading should be at least 10 megohms/cm. Record in log book. (16 megohms/cm maximum load).

Michael J. Linskens
Michael J. Linskens
Director, Technical Services

Kim D. Finner
Kim D. Finner
Laboratory Manager

Revision Date

8-18-87

2-5-90